



Electron Diffraction

Applications Using the
PDF-4+ Relational Database

Electron Diffraction in Materials Science

- Electron diffraction is an important technique for crystallographic characterization, a valuable complementary tool to powder and single crystal X-ray diffraction.
- Applications include phase identification and precision determination of subtle structural details for crystals in the micrometer to nanometer size range.

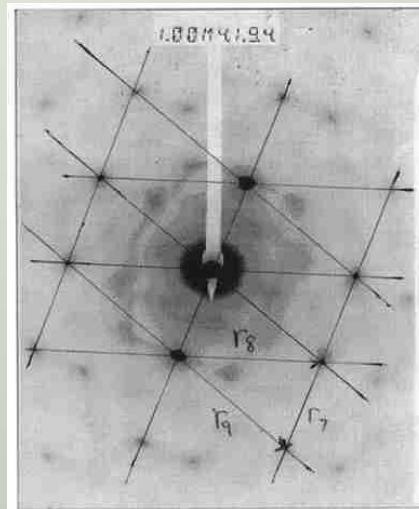
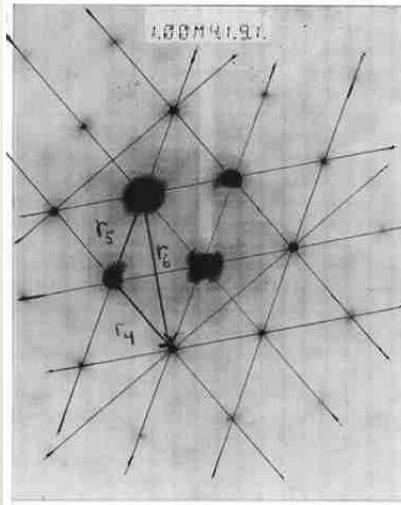
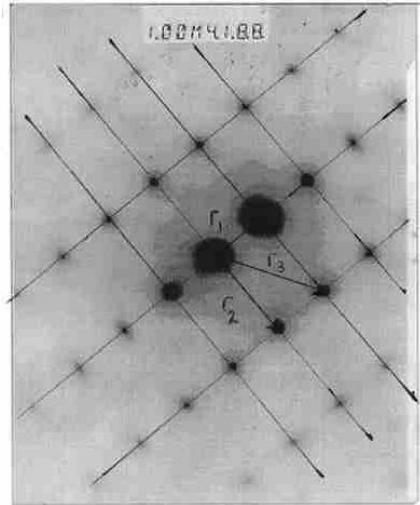
Electron Diffraction with the PDF-4+ Database

- The PDF-4+ database can be used to generate selected area electron diffraction (SAED) and electron backscatter diffraction (EBSD) patterns. This tutorial will deal with SAED patterns.
- The SAED patterns simulated by PDF-4+ 2009 use calculated intensities (using atomic scattering factors for electrons) when atomic coordinates are available, and intensities tabulated in the PDF entries when atomic coordinates are unavailable.

SAED Patterns with the PDF-4+ Database

- The PDF-4+ 2009 database does not perform search-match procedures directly on digital SAED patterns. They must first be indexed to obtain a D-spacing-Intensity (D-I) list, then search-match procedures can be performed using Sleave+.
- Obtaining a D-I list and performing search-match using Sleave+ will be illustrated in the following slides.

Prepare a D-I List



- Use SAED patterns obtained from multiple zone axes with a known camera constant to prepare a D-I list:

d (Å)	I/I _o
2.086	100
1.277	50
1.089	50
0.829	50

Use All Available Information

- The current sample is a metal alloy containing iron (based on EDS analysis).

- Before performing search-match with the D-I list in Slever+, search the PDF database for compounds meeting the sample criteria:
 - Empirical formula contains 'Fe'
 - Subfile: Metals and Alloys

Searching for Iron Alloys

Results - {Subfile/Subclass (Metals ...}

File Edit Fields Results Indexing Help

Results (10,504 of 291,440)

Search Preference Set: ICDD Defaults

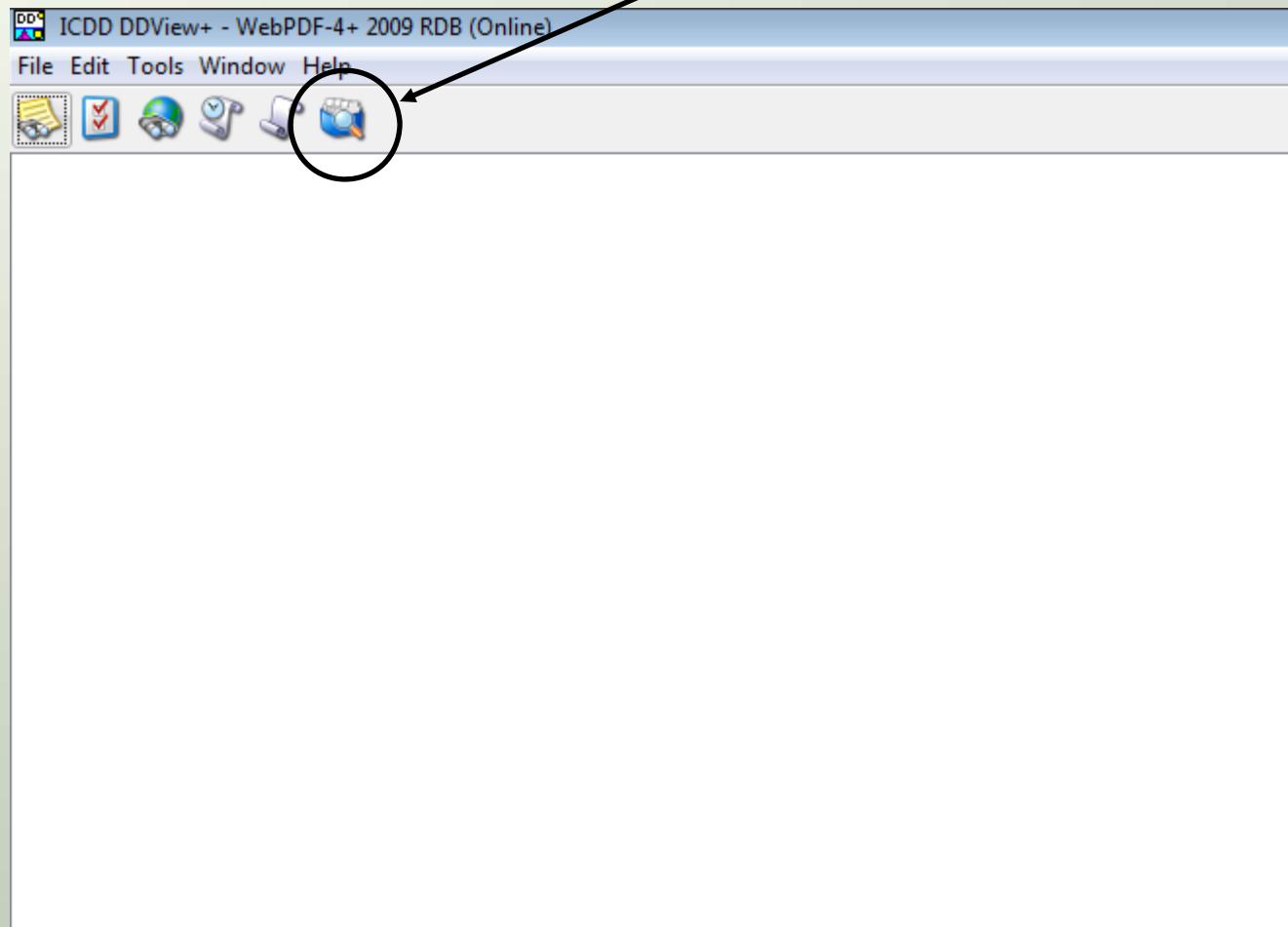
PDF #	QM	Chemical Formula	Mineral Name	SYS	SPGR	SG #	AuthC
00-001-0842	B	Cu Fe S2	Chalcopyrite	T	I-42d	122	4.00
00-001-1053	I	Fe2 O3	Hematite	R	R-3c	167	6.00
00-001-1111	B	Fe3 O4	Magnetite	C	Fd-3m	227	8.00
00-001-1200	B	Fe2 P		H	P321	150	3.00
00-001-1219	B	Fe4 N		C			1.00
00-001-1223	B	Fe O		C	Fm-3m	225	4.00
00-001-1228	I	Fe2 Al5		M			
00-001-1236	B	Fe3 N		H	P63/mmc	194	0.75
00-001-1247	B	Fe S	Troilite	H	P63/mmc	194	2.00
00-001-1252	O	Fe		C	Im-3m	229	2.00
00-001-1257	B	Al Fe		C	Pm-3m	221	1.00
00-001-1262	B	Fe		C	Im-3m	229	2.00
00-001-1265	O	Fe Al3		X			
00-001-1267	B	Fe		C	Im-3m	229	2.00
00-001-1271	B	Fe Si		C	P213	198	4.00
00-001-1285	B	Fe Si2		T	P4/m	83	1.00
00-001-1295	B	Fe S2	Pyrite	C	Pa-3	205	4.00
00-002-0264	I	Ag Fe2 S3	Sternbergite	O	Ccomm	63	8.00
00-002-0426	S	Sb6 Fe Pb4 S14	Jamesonite	M	P21/a	14	2.00
00-002-0500	B	Cu Fe2 S3	Cubanite	O	Pnma	62	4.00

Search Description: {Subfile/Subclass (Metals & Alloys)} And {Empirical Formula Contains Elements 'Fe'}

Calculations: Mean: Median: ESD:

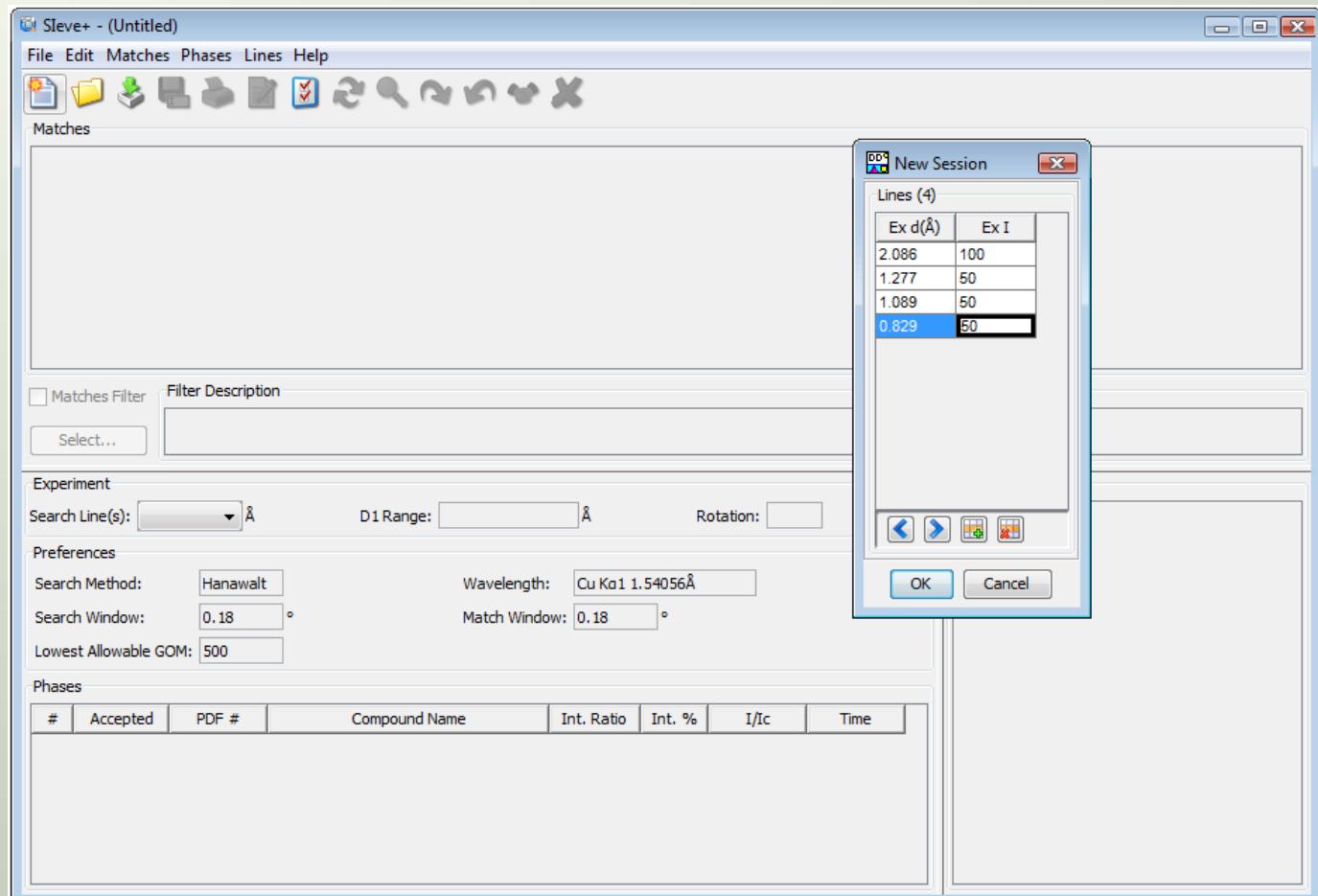
Using Sleeve+

Begin a Sleeve+ session by pressing the 'Sleeve+' button...



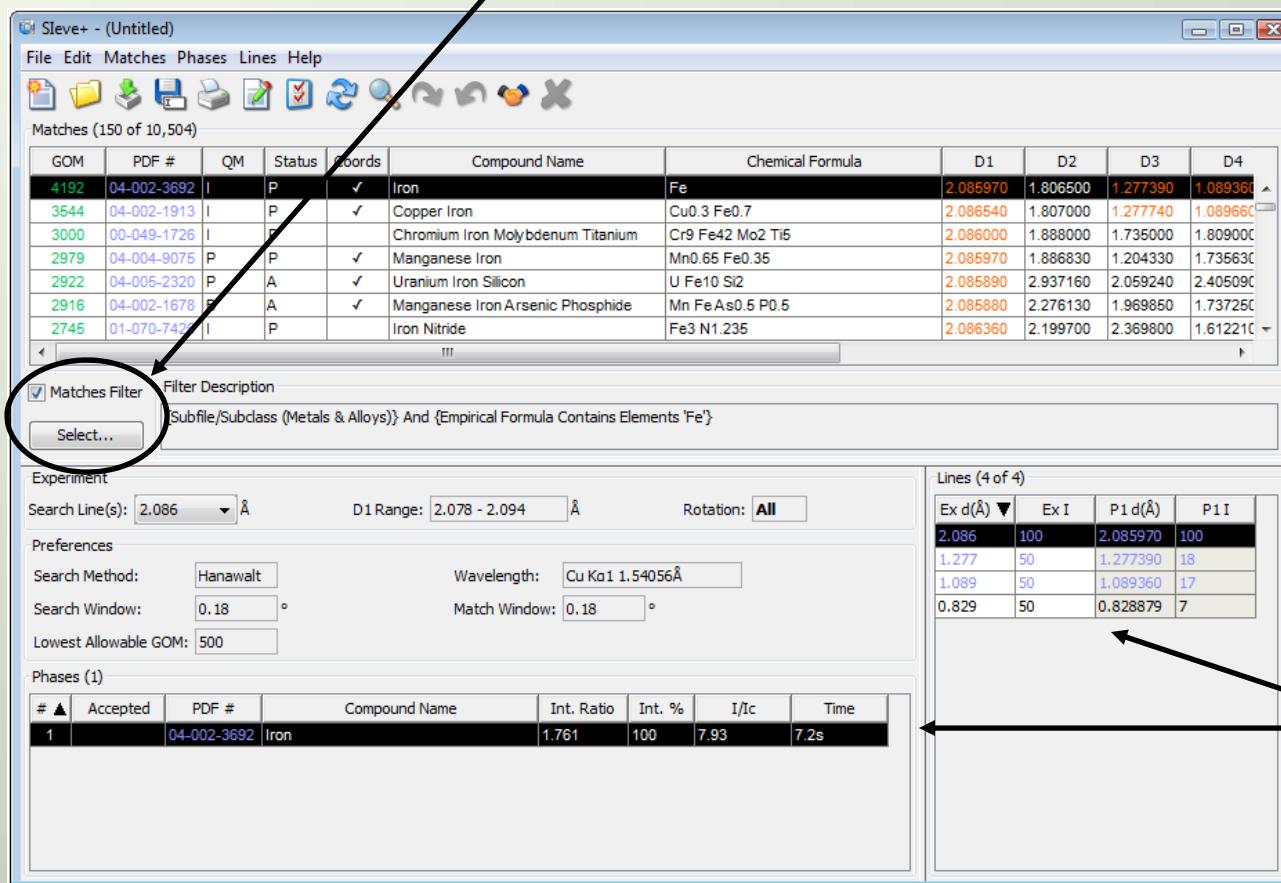
Using Sleve+

...then choose 'File → New Session' from the pull down menu, input the D-I list, and press the 'OK' button to begin search-match.



Adding the Search Results

Check the ‘Matches Filters’ box and choose the search for alloys containing Fe to limit the search space.



The screenshot shows the Slever+ software interface with the following details:

- Matches (150 of 10,504)**: A table listing search results. The first few rows include:

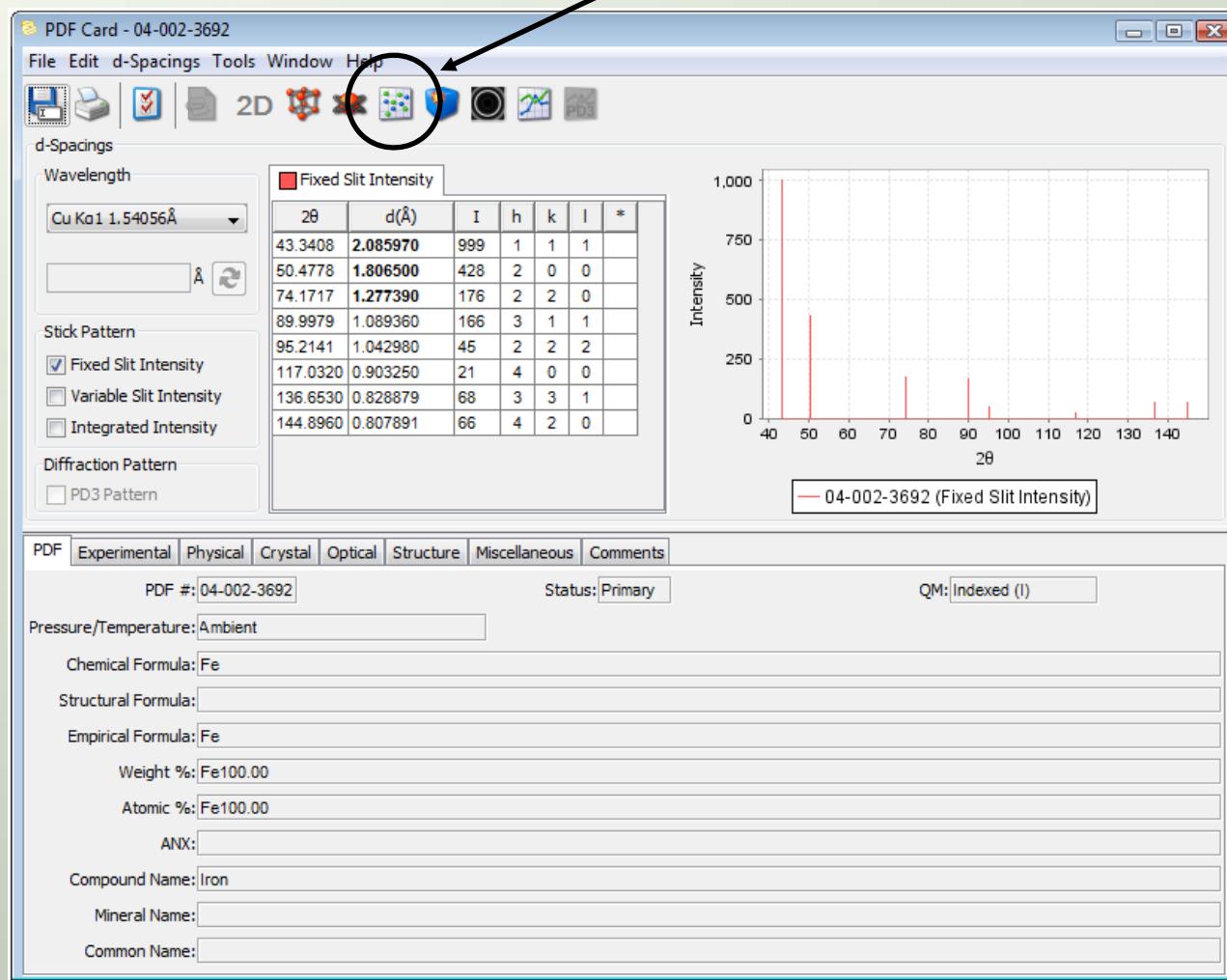
GOM	PDF #	QM	Status	Boards	Compound Name	Chemical Formula	D1	D2	D3	D4
4192	04-002-3692	I	P	✓	Iron	Fe	2.085970	1.806500	1.277390	1.089360
3544	04-002-1913	I	P	✓	Copper Iron	Cu0.3 Fe0.7	2.086540	1.807000	1.277740	1.089660
3000	00-049-1726	I	P		Chromium Iron Molybdenum Titanium	Cr9 Fe42 Mo2 Ti5	2.086000	1.888000	1.735000	1.809000
2979	04-004-9075	P	P	✓	Manganese Iron	Mn0.65 Fe0.35	2.085970	1.886830	1.204330	1.735630
2922	04-005-2320	P	A	✓	Uranium Iron Silicon	U Fe10 Si2	2.085890	2.937160	2.059240	2.405090
2916	04-002-1678	P	A	✓	Manganese Iron Arsenic Phosphide	Mn Fe As0.5 P0.5	2.085850	2.276130	1.969850	1.737250
2745	01-070-742	I	P		Iron Nitride	Fe3 N1.235	2.086360	2.199700	2.369800	1.612210
- Filter Description**: Subfile/Subclass (Metals & Alloys) And {Empirical Formula Contains Elements 'Fe'}
- Experiment** section: Search Line(s): 2.086 Å, D1 Range: 2.078 - 2.094 Å, Rotation: All
- Preferences** section: Search Method: Hanawalt, Wavelength: Cu Kα1 1.54056 Å, Search Window: 0.18, Match Window: 0.18, Lowest Allowable GOM: 500
- Phases (1)**: Accepted PDF #: 04-002-3692, Compound Name: Iron, Int. Ratio: 1.761, Int. %: 100, I/Ic: 7.83, Time: 7.2s
- Lines (4 of 4)**: A table showing peak positions. The first row is highlighted in yellow:

Ex d(Å)	Ex I	P1 d(Å)	P1 I
2.086	100	2.085970	100
1.277	50	1.277390	18
1.089	50	1.089360	17
0.829	50	0.828879	7

In this case,
we have a
very good
match for
Iron (FCC).

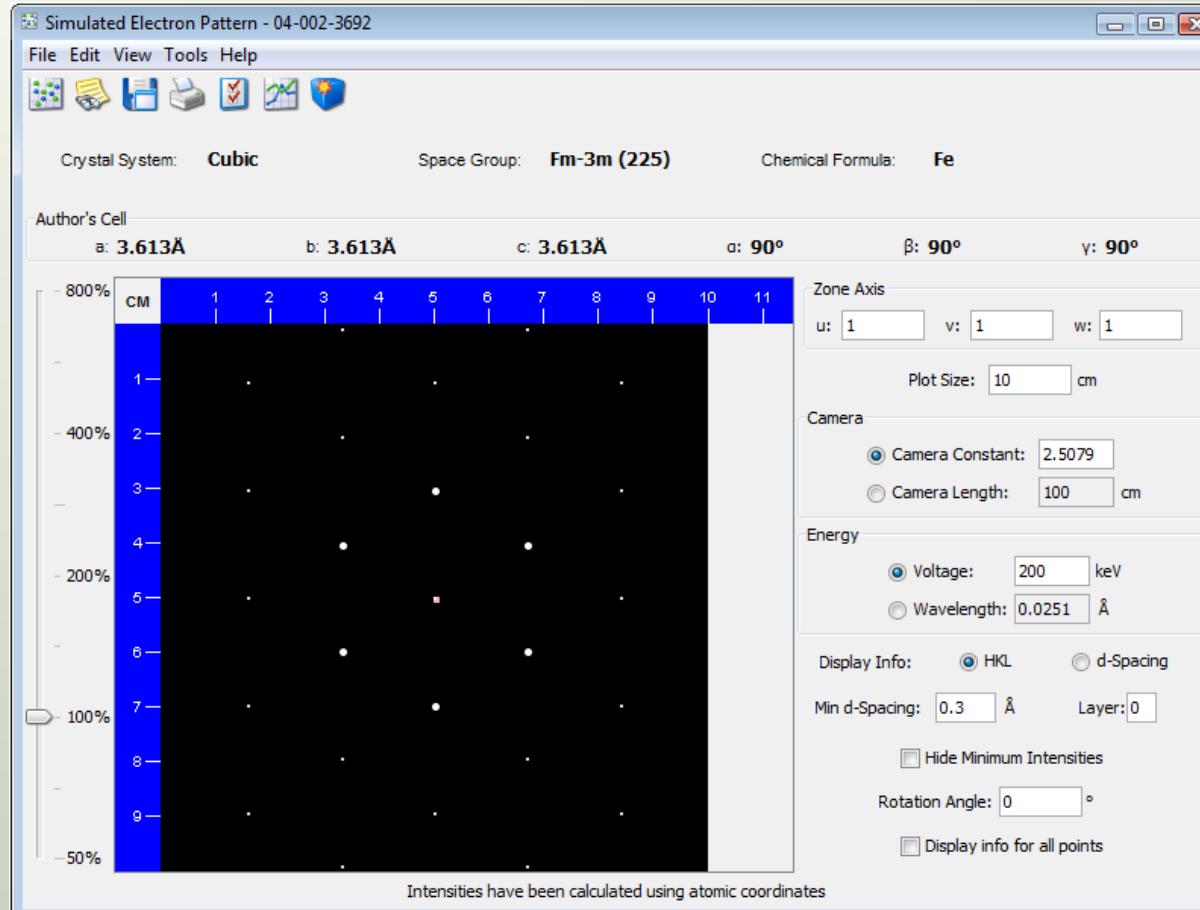
Accessing SAED Patterns

Press the 'SAED Pattern' button...



SAED Patterns

...and the Simulated Electron Pattern window will appear.



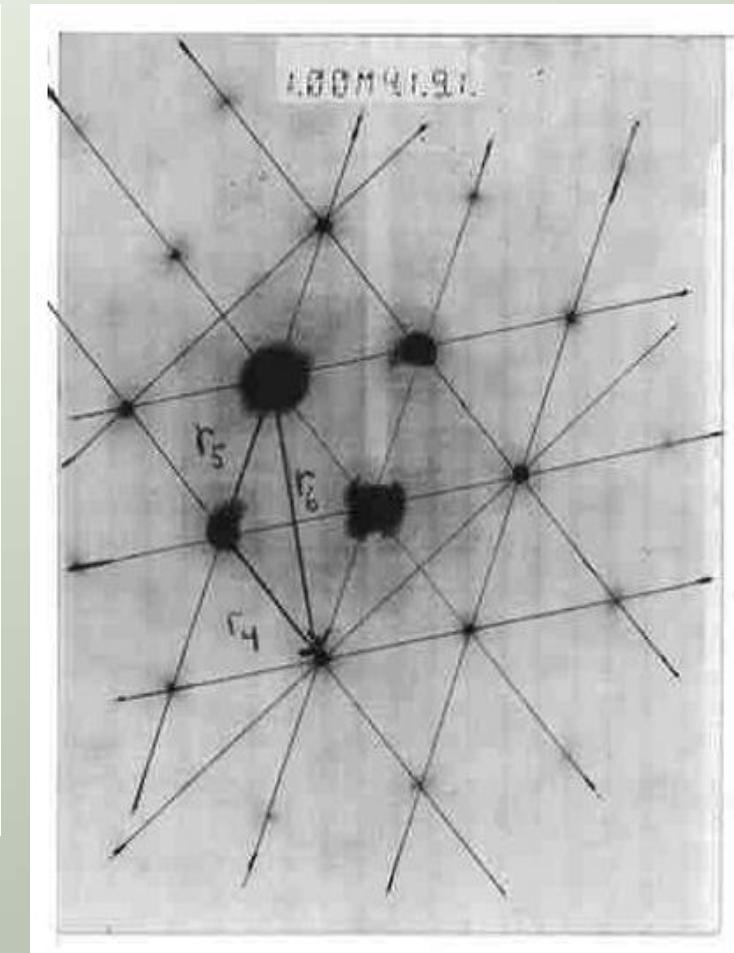
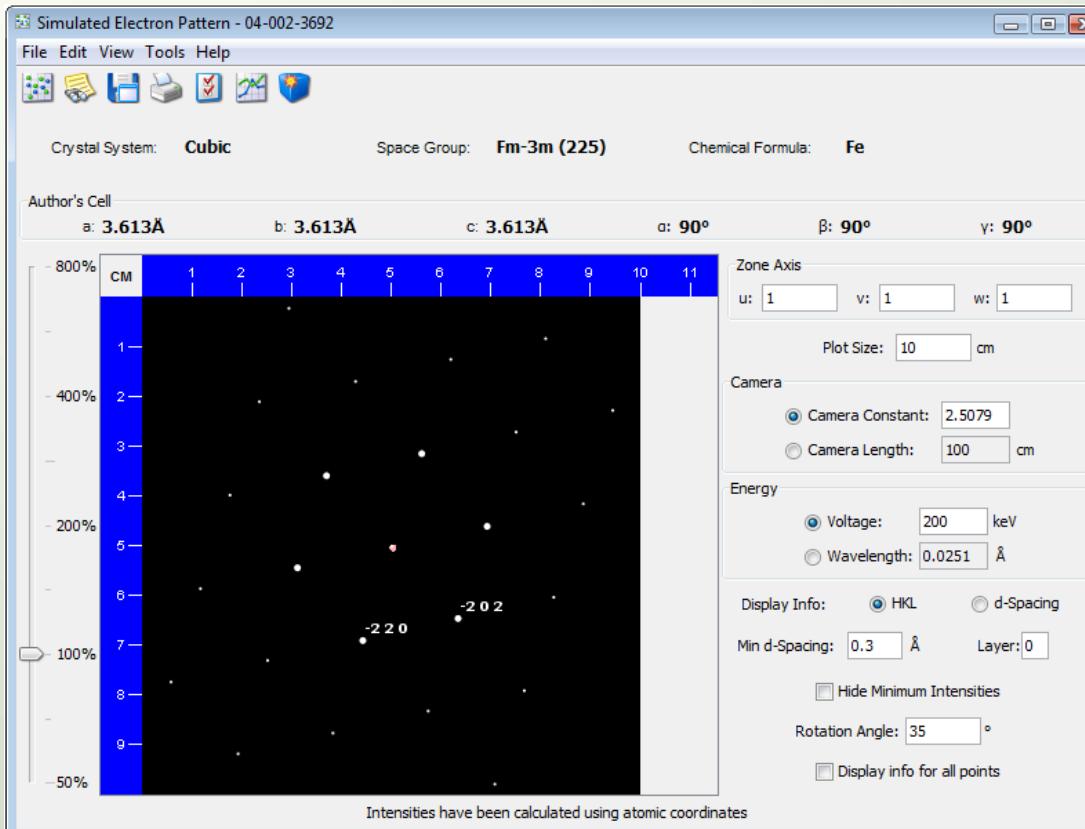
Preferences such as the zone axis, camera constant, and accelerating voltage can be edited with on-the-fly dynamic re-simulation of the pattern.

SAED Patterns for Iron – Simulation & Experiment

- The following three slides compare simulated and experimental SAED patterns for FCC Iron (Fe, space group Fm-3m) for three zone axes using PDF card 04-002-3692, which was one of the top two matches to the D-I list using search-match with Slever+.

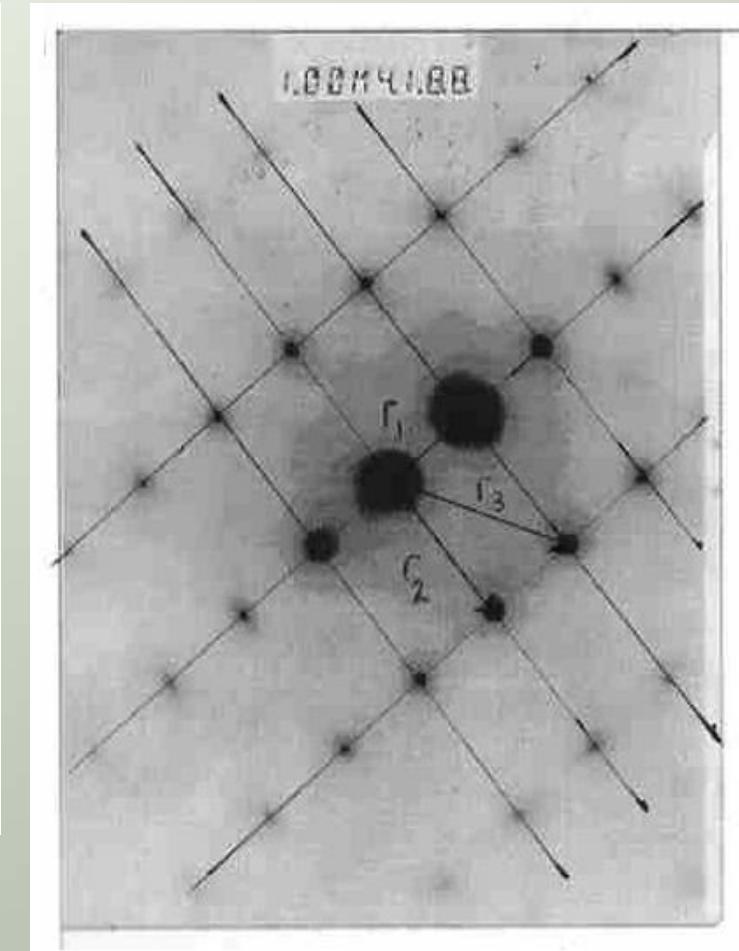
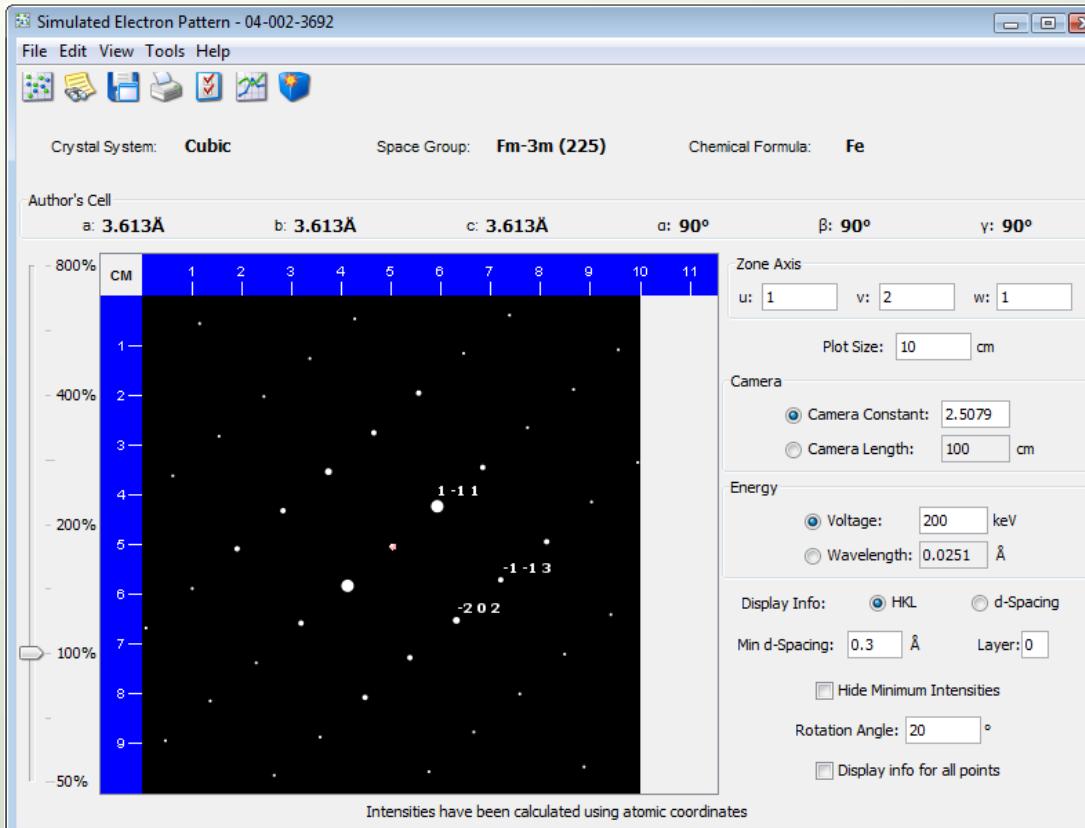
FCC Iron (Fe, Fm-3m)

[111] Zone Axis



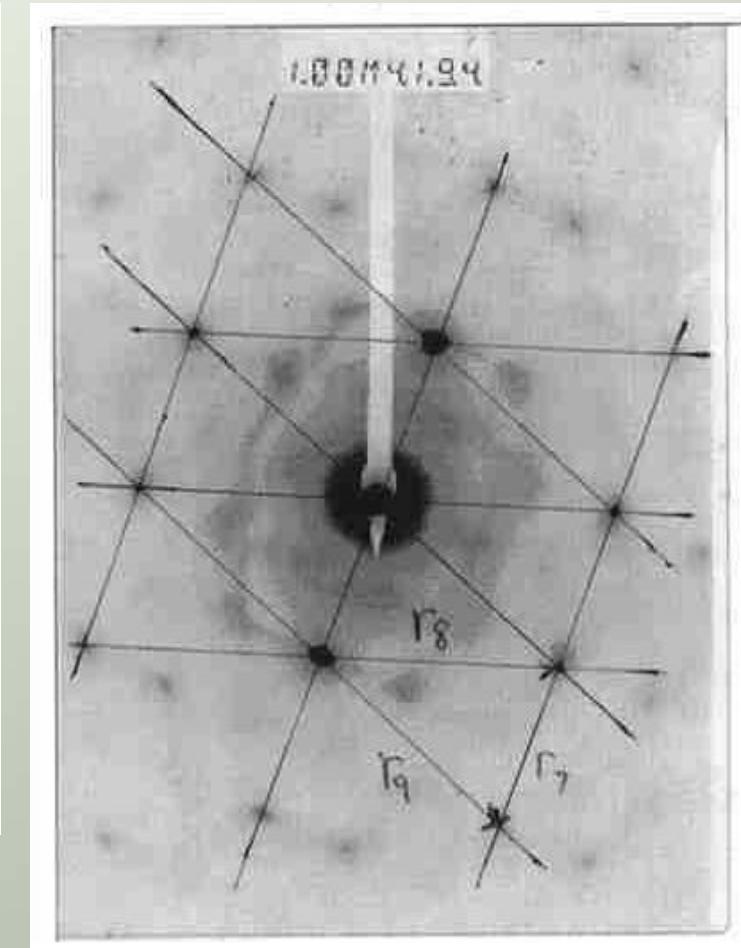
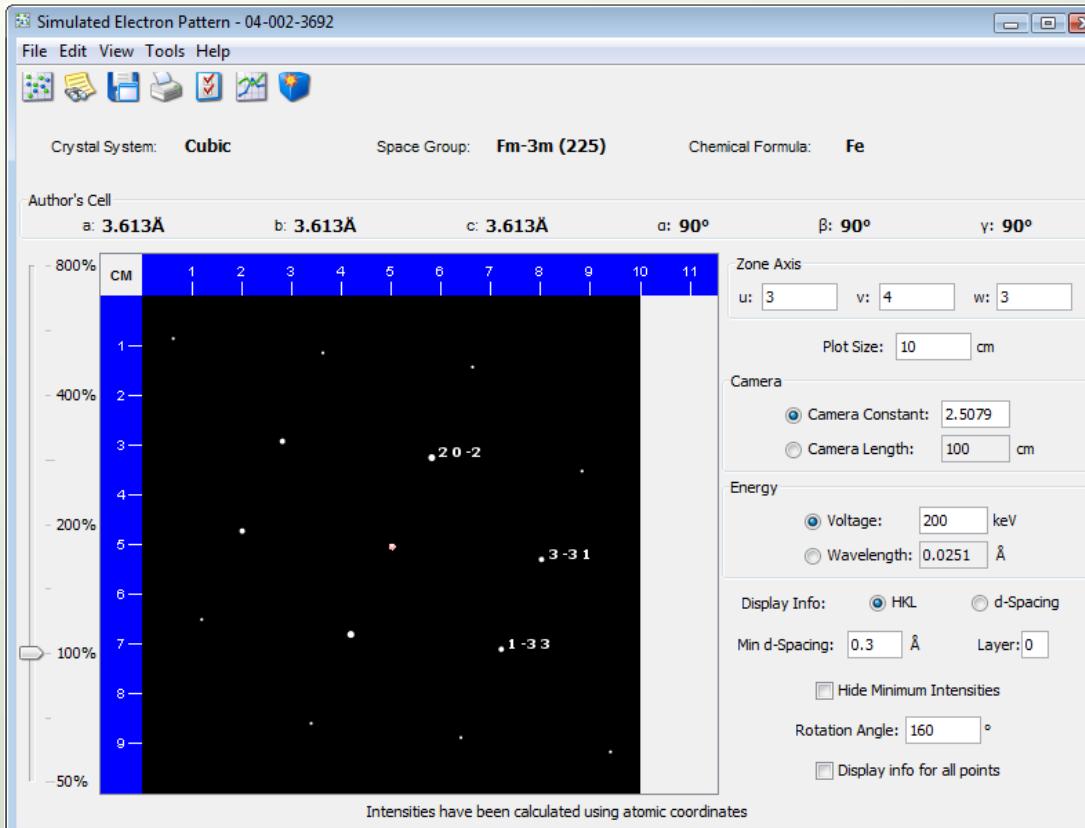
FCC Iron (Fe, Fm-3m)

[121] Zone Axis



FCC Iron (Fe, Fm-3m)

[343] Zone Axis



SAED Patterns – Summary

- The simulated FCC Iron SAED patterns match closely with the experimental patterns.
- PDF-4+ 2009 generates SAED patterns with intensities calculated from atomic coordinates based on atomic scattering factors for electrons when a structural model is available, and SAED patterns with intensities approximated using the D-I list for entries without atomic coordinates.



Thank you for viewing our tutorial.
Additional tutorials are available at the ICDD website
(www.icdd.com).



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