

# **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 Sleve+.
- Obtaining a D-I list and performing search-match using Sleve+ will be illustrated in the following slides.



#### Prepare a D-I List





1.88M41.94

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

d (Å)	۱/۱ <sub>o</sub>
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 Sleve+, search the PDF database for compounds meeting the sample criteria:
  - Empirical formula contains 'Fe'
  - Subfile: Metals and Alloys



# Searching for Iron Alloys

Results (10,504 Search Prefere PDF # 00-001-0842 E 00-001-1053 I 00-001-1111 E 00-001-1200 E	4 of 291,440) nce Set: [CC QM 3 Cu F	DD Defaults Chemical Formula	Mineral Name				
PDF #         PDF #           00-001-0842         E           00-001-1053         I           00-001-1111         E           00-001-1200         E	QM QM B Cu F	DD Defaults Chemical Formula	Mineral Name				
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00-001-0842 E 00-001-1053 I 00-001-1111 E 00-001-1200 E	B Cu F	- 00	Thirter or Frontie	SYS	SPGR	SG #	Auth
00-001-1053   00-001-1111   E 00-001-1200   E	Ee2	e 52	Chalcopyrite	Т	I-42d	122	4.00
00-001-1111 E		O3	Hematite	R	R-3c	167	6.00
00-001-1200 E	B Fe3	04	Magnetite	С	Fd-3m	227	8.00
	B Fe2	P		Н	P321	150	3.00
00-001-1219 E	B Fe4	N		С			1.00
00-001-1223 E	B Fe O	)		С	Fm-3m	225	4.00
00-001-1228	Fe2/	A15		M			
00-001-1236 E	B Fe3	N		Н	P63/mmc	194	0.75
00-001-1247 E	B Fe S		Troilite	Н	P63/mmc	194	2.00
00-001-1252 O Fe C Im-3m 229 2.00							
00-001-1257 B AI Fe C Pm-3m 221 1.00							
00-001-1262 E	B Fe			С	lm-3m	229	2.00
00-001-1265	D FeA	13		X			
00-001-1267 E	3 Fe			С	lm-3m	229	2.00
00-001-1271 E	B Fe S	i		С	P213	198	4.00
00-001-1285 E	3 Fe S	12		Т	P4/m	83	1.00
00-001-1295 E	3 Fe S	2	Pyrite	С	Pa-3	205	4.00
00-002-0264	Ag F	e2 S3	Sternbergite	0	Ccmm	63	8.00
00-002-0426 5	S Sb6	Fe Pb4 S14	Jamesonite	М	P21/a	14	2.00
00-002-0500 E	B Cu F	e2 S3	Cubanite	0	Pnma	62	4.00
•		III		1	1	1	
Search Descript	tion		Calcul	ations			



#### **Using Sleve+**

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

ICDD DDView+ - WebPDF-4+ 2009 RDB (Online)
File Edit Tools Window Help



#### **Using Sleve+**

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

🖏 SIeve+ - (Untitled)	
File Edit Matches Phases Lines Help	
	New Session           Lines (4)           Ex d(Å)         Ex I           2.086         100           1.277         50           1.089         50           0.829         50
Matches Filter Description	
Experiment Search Line(s): <ul> <li>Å</li> <li>D1 Range:</li> <li>Å</li> <li>Rotation:</li> </ul>	
Search Method:     Hanawalt     Wavelength:     Cu Kol 1.54056Å       Search Window:     0.18     °       Lowest Allowable GOM:     500	OK Cancel
Phases	
#         Accepted         PDF #         Compound Name         Int. Ratio         Int. %         I/Ic         T	Time



# Adding the Search Results

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

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		~ /	0.4 0. 0 00					
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GOM PDF # Q	4 Status	Coords	Compound Name	Chemical Formula	D1	D2	D3	D4
4192 04-002-3692 I	P	1	Iron	Fe	2.085970	1.806500	1.277390	1.089360 🔺
3544 04-002-1913 I	P	1	Copper Iron	Cu0.3 Fe0.7	2.086540	1.807000	1.277740	1.089660
3000 00-049-1726 I			Chromium Iron Molybdenum Titanium	Cr9 Fe42 Mo2 Ti5	2.086000	1.888000	1.735000	1.809000
2979 04-004-9075 P	P	1	Manganese Iron	Mn0.65 Fe0.35	2.085970	1.886830	1.204330	1.735630
2922 04-005-2320 P	A	1	Uranium Iron Silicon	U Fe10 Si2	2.085890	2.937160	2.059240	2.405090
2916 04-002-1678 F	A	1	Manganese Iron Arsenic Phosphide	Mn Fe As0.5 P0.5	2.085880	2.276130	1.969850	1.737250
2745 01-070-7427 1	P		Iron Nitride	Fe3 N1.235	2.086360	2.199700	2.369800	1.612210 -
Matches Filter Filter Desc Select	iption bclass (Metal	s & Alloys	)} And {Empirical Formula Contains Eleme	ents 'Fe'}				
Experiment					Lines (4 of 4	.)		
earch Line(s): 2.086	8	D1R	ange: 2 078 - 2 094 Å	Rotation: All	Ex d(Å) ▼	- Fy I	P1 d(Å)	PIT
	-	DIR			2.086	100	2 085970 1	100
Preferences					1.277	50	1.277390 1	18
Search Method: Hana	valt		Wavelength: Cu Ko1 1.540	056Å	1.089	50	1.089360 1	L7
Search Window: 0.18	۰		Match Window: 0.18	>	0.829	50	0.828879 7	7
Lowest Allowable GOM: 500								
Phases (1)								
# Accopted DDE #		Comp	und Name Int Datio In	+ 9/ I/Ic Time				
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1 04-002-36	92 Iron		1.761 100	J 7.93 7.2s				
								4

n this case, we have a very good match for ron (FCC).



# **Accessing SAED Patterns**

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	hutton					
					button	
PDF Card - 04-002-3692					- 0 🔀	
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d-Spacings	$\bigcirc$					
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Å 🎅	50.4778 <b>1.806500</b>	428 2 0	0	sity		
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Variable Slit Intensity	136.6530 0.828879	68 3 3	1			
Integrated Intensity	144.8960 0.807891	66 4 2	0			
Diffraction Pattern					20	
PD3 Pattern					-04-002-3692 (Fixed Slit Intensity)	
		1	1	1		
PDF Experimental Physical C	Crystal Optical Structu	re Miscellaneous	Comments			
PDF #: 04-002-3	692	Stat	tus: Primary		QM: Indexed (I)	
Pressure/Temperature: Ambient						
Chemical Formula: Fe						
Structural Formula:						
Empirical Formula: Fe						
Weight %: Fe100.00	)					
Atomic %: Fe100.00	)					
ANX:						
Compound Name: Iron						
Mineral Name:						
Common Name:						



#### **SAED** Patterns

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





### 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 Sleve+.



## FCC Iron (Fe, Fm-3m) [111] Zone Axis

🔠 Simulated Electron Pattern - 04-00	2-3692			- • •
File Edit View Tools Help				
🔢 😂 📑 🍛 🗵 🖄	<b>9</b>			
Crystal System: Cubic	Space Group	p: Fm-3m (225)	Cher	nical Formula: Fe
Author's Cell a: 3.613Å b	3.613Å	c: <b>3.613Ă</b>	a: <b>90°</b>	β: <b>90°</b> γ: <b>90°</b>
- <sup>800%</sup> CM 1 2	3456         •	789 	10 11 	Zone Axis u: 1 v: 1 w: 1
1-				Plot Size: 10 cm
- 400% 2-				Camera Constant: 2.5079
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4	•	•		<ul> <li>energy</li> <li>Ovoltage: 200 keV</li> <li>Ovoltaget: 0.0251 Å</li> </ul>
6 - · · · · · · · · · · · · · · · · · ·	-220	-202		Display Info:
8-				Hide Minimum Intensities
9 — . _50%				Display info for all points
	Intensities hav	ve been calculated using at	comic coordina	ates





## FCC Iron (Fe, Fm-3m) [121] Zone Axis

🔯 Simulated Electron Pa	ttern - 04-002-3692			- • •
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10 😓 🌏 🔝	M 🖗			
Crystal System: C	Cubic	Space Group: Fm-3m (225)	Che	mical Formula: Fe
Author's Cell a: <b>3.613Ă</b>	b: 3.613Å	c: <b>3.613</b> Å	a: <b>90</b> °	β: <b>90°</b> γ: <b>90°</b>
<sup>-800%</sup> cm 1	2 3 4 	56789 	10 11 	Zone Axis u: 1 v: 2 w: 1
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3-	· •	•		Camera Length: 100 cm
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- 200% 5	•	• •		Wavelength: 0.0251 Å
- <mark>6-</mark> ·	•	-202		Display Info: 💿 HKL 💿 d-Spacing
- 100% <sup>7-</sup>		•		Min d-Spacing: 0.3 Å Layer: 0
8-		, ·		Hide Minimum Intensities
9— •				Rotation Angle: 20 °
-50%	•			Display info for all points
		Intensities have been calculated using a	tomic coordin	ates





## FCC Iron (Fe, Fm-3m) [343] Zone Axis

File Edit View Tools Help         Image: Solution of the solution o	Simulated Electron Pattern - 04-002-369.	2		- • •
Crystal System: Cubic Space Group: Fm-3m (225) Chemical Formula: Fe Author's Cell a: 3.613Å b: 3.613Å c: 3.613Å c: 3.613Å c: 90° ß: 90° y: 90° CM 1 2 3 4 5 6 7 0 0 10 11 Cone Axis u: 3 v: 4 w: 3 Flot Size: 10 m Camera Camera Constant: 2.5079 Camera Length: 100 cm Energy Voltage: 200 keV Wavelength: 0.0251 Å Display Info: HKL d'Spacing Min d-Spacing: 0.3 Å Layer:0 HKL d'Spacing Biological Ministres Rotation Angle: 160 ° Display info for all points	File Edit View Tools Help			
Crystal System:       Cubic       Space Group:       Fm-3m (225)       Chemical Formula:       Fe         Author's Cell <ul> <li>a. 3.613Å</li> <li>b: 3.613Å</li> <li>c: 90°</li> <li>p: 90°</li> <li>y: 90°</li></ul>	🗟 🍪 🍯 🧽 🚺			
Author's Cell       a:       3.613Å       b:       3.613Å       c:       3.613Å       c:       90°       β:       90°       γ:       90°         800%       CM       1       2       3       4       6       0       7       6       10       11         400%       2       -       -       -       -       0       0       10       rd         200%       2       -       -       -       -       0       -	Crystal System: Cubic	Space Group: Fm-3m (225)	Chemical Formula:	Fe
800%       CM       1       2       3       4       5       6       7       8       9       10       11         400%       1 <t< td=""><td>Author's Cell a: 3.613Å b: 3.61</td><td>L3Ă c: 3.613Ă</td><td>a: <b>90</b>° (i</td><td>3: <b>90°</b> γ: <b>90°</b></td></t<>	Author's Cell a: 3.613Å b: 3.61	L3Ă c: 3.613Ă	a: <b>90</b> ° (i	3: <b>90°</b> γ: <b>90°</b>
000 /s       CM       1       2       3       4       6       6       7       8       6       10       11         400%       2       -         Plot Size: 10       0         3            Camera Constant: 2.5079          3            Camera Length: 100       cm         4                 200%       6 <td>800%</td> <td></td> <td>Zone Avis</td> <td></td>	800%		Zone Avis	
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- 200%       -       .3 - 3 1          ⓐ Voltage: 200 keV         ⓑ Wavelength: 0.0251 Â         〕         ⓑ Display Info: ⓐ HKL ⓓ d-Spacing         ⓑ HKL ⓓ d-Spacing         ⓑ HKL ⓓ d-Spacing         ⓑ Hide Minimum Intensities         Rotation Angle: 160 °         ⓑ Display info for all points			Energy	
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0       0	5-	* .3-31		Wavelength: 0.0251 Å
6-       Display Info:        HKL       d-Spacing         7-       .1-3 3       Min d-Spacing:       0.3 Å       Layer:       0         8-       Image: Hide Minimum Intensities       Rotation Angle:       160 °       0         -50%       Interstities have been calculated using atomic coordinates       Display info for all points				
7-       1-33       Min d-Spacing: 0.3 Å Layer: 0         8-       Hide Minimum Intensities         9-       Rotation Angle: 160 °         -50%       Display info for all points	6-		Display Info	● HKL ◎ d-Spacing
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# 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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