Data Mining Tools

Sorted Displays
Histograms
Sleve

Data Mining
What?

From Webopedia:
A class of database applications that look for hidden patterns in a group of data that can be used to predict future behavior. The term is commonly misused to describe software that presents data in new ways.
True data mining software doesn't just change the presentation, but actually discovers previously unknown relationships among the data.

From Wikipedia:
Data mining has been defined as "the nontrivial extraction of implicit, previously unknown, and potentially useful information from data" and "the science of extracting useful information from large data sets or databases".

Data Mining

Why?

The Powder Diffraction File (PDF) contains diffraction, crystallographic, bibliographic, and physical property information on ~550,000 unique entries. This is the world's largest collection of structural and physical property information on solid states materials.

Data mining can help scientist discover new information on how materials work.

Data Mining

How?

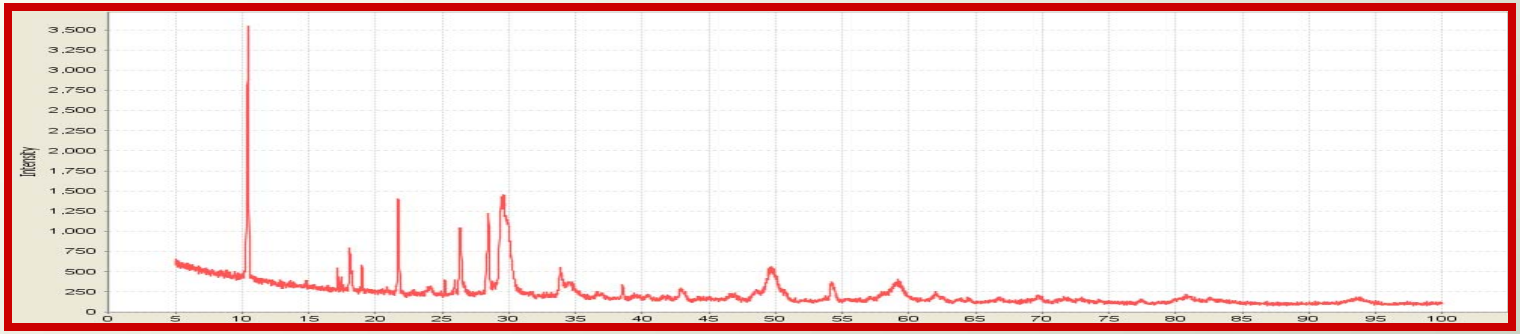
The Powder Diffraction File allows users to search by 48 different search mechanisms and then display data using 66 fields.

The fields and display searches can be combined and sorted – providing almost limitless data mining combinations.

Data can be graphed in xy plots or histograms.

Multiple cards or diffraction patterns can be displayed simultaneously.

Data Mining



This is the diffraction pattern taken from a specimen of a catalytic converter. In this tutorial, this pattern will be extensively analyzed.

From this diffraction pattern you can identify the bulk composition and quantitate the results using modern X-ray analysis techniques.

Using data mining of the PDF-4 database, you can determine solid solution doping, the temperature of synthesis for the part, and make reasonable assumptions about the identity and concentration of low concentration catalysts present in the specimen.

From the above data, you can find applicable patents on the Internet that align with the data and describe the manufacture of the part. Overall data mining leads to a reasonable hypothesis for the fabrication of the component and its exact composition that could be verified, if required, with further experimentation.

Example

Data were collected on a commercial catalytic converter.
Very little information is known about the specimen.

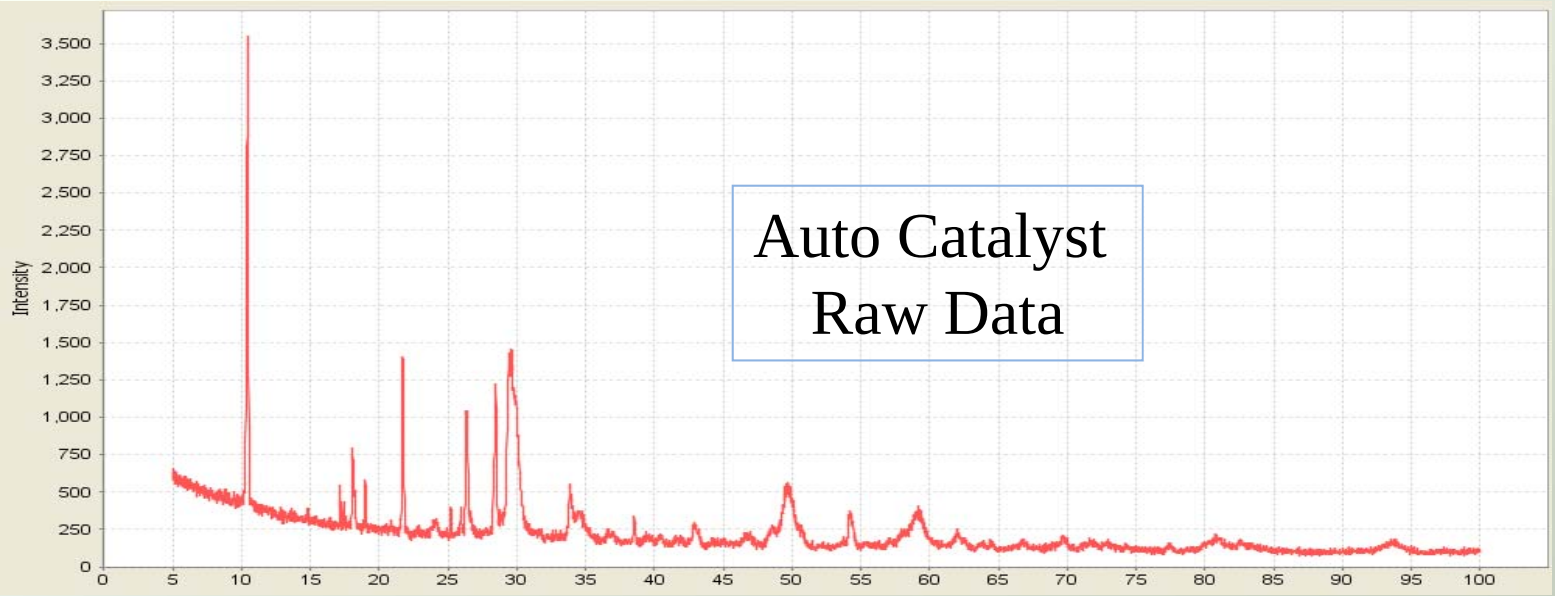
The sequence is

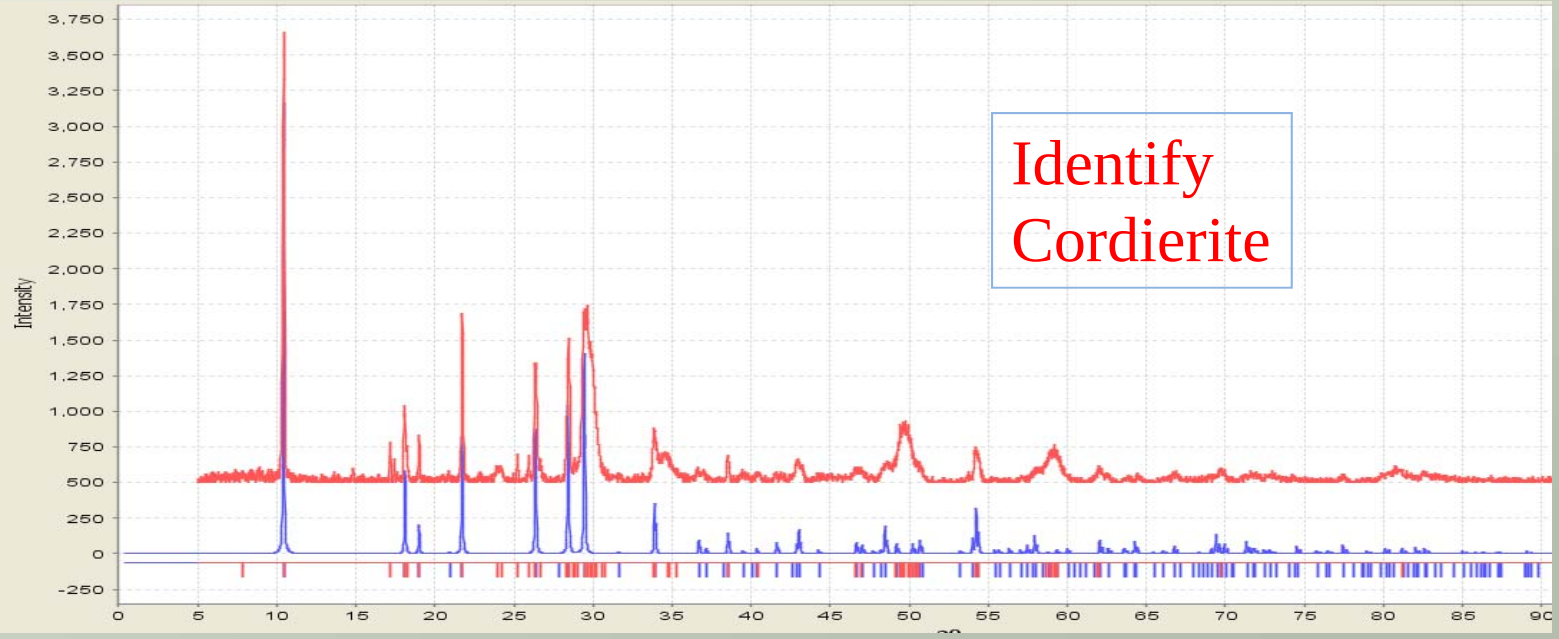
- Phase identification
- Quantitation
- Unit cell refinement

then

- Data mining based on the above experimentally defined parameters

The next few slides outline the sequence of events required to solve the problem. For step-by-step procedures see the tutorials on SIeve+


Intensity
3,500
3,250
3,000
2,750
2,500
2,250
2,000
1,750
1,500
1,250
1,000
750
500
250
0
0 5 10 15 20 25 30 35 40 45 50 55 60 65 70 75 80 85 90 95 100
Auto Catalyst
Raw Data


Intensity
3,750
3,500
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3,000
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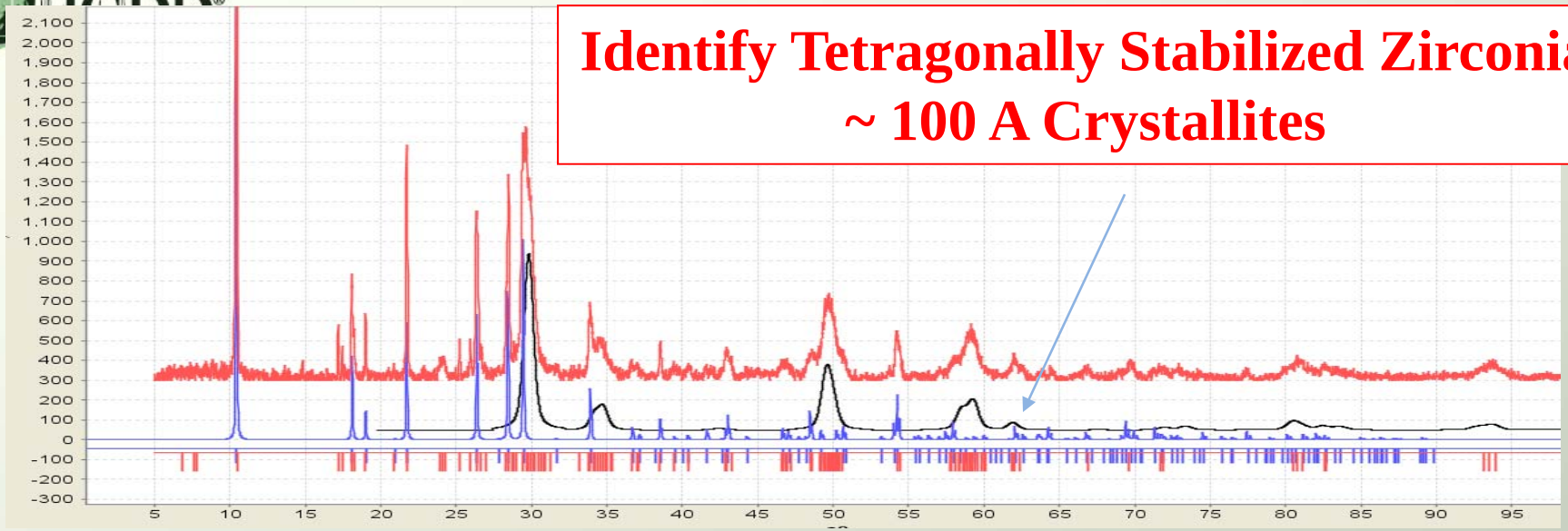
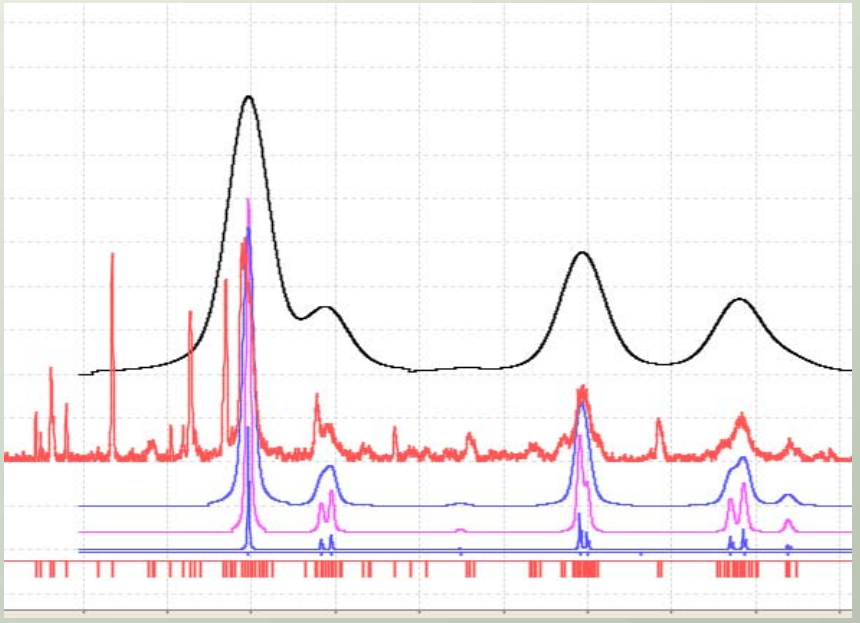
2,000
1,750
1,500
1,250
1,000
750
500
250
0
-250
0 5 10 15 20 25 30 35 40 45 50 55 60 65 70 75 80 85 90
Identify
Cordierite

Identify Tetragonally Stabilized Zirconia ~ 100 A Crystallites





Identify the size through zirconia pattern simulations

25 A

Experimental

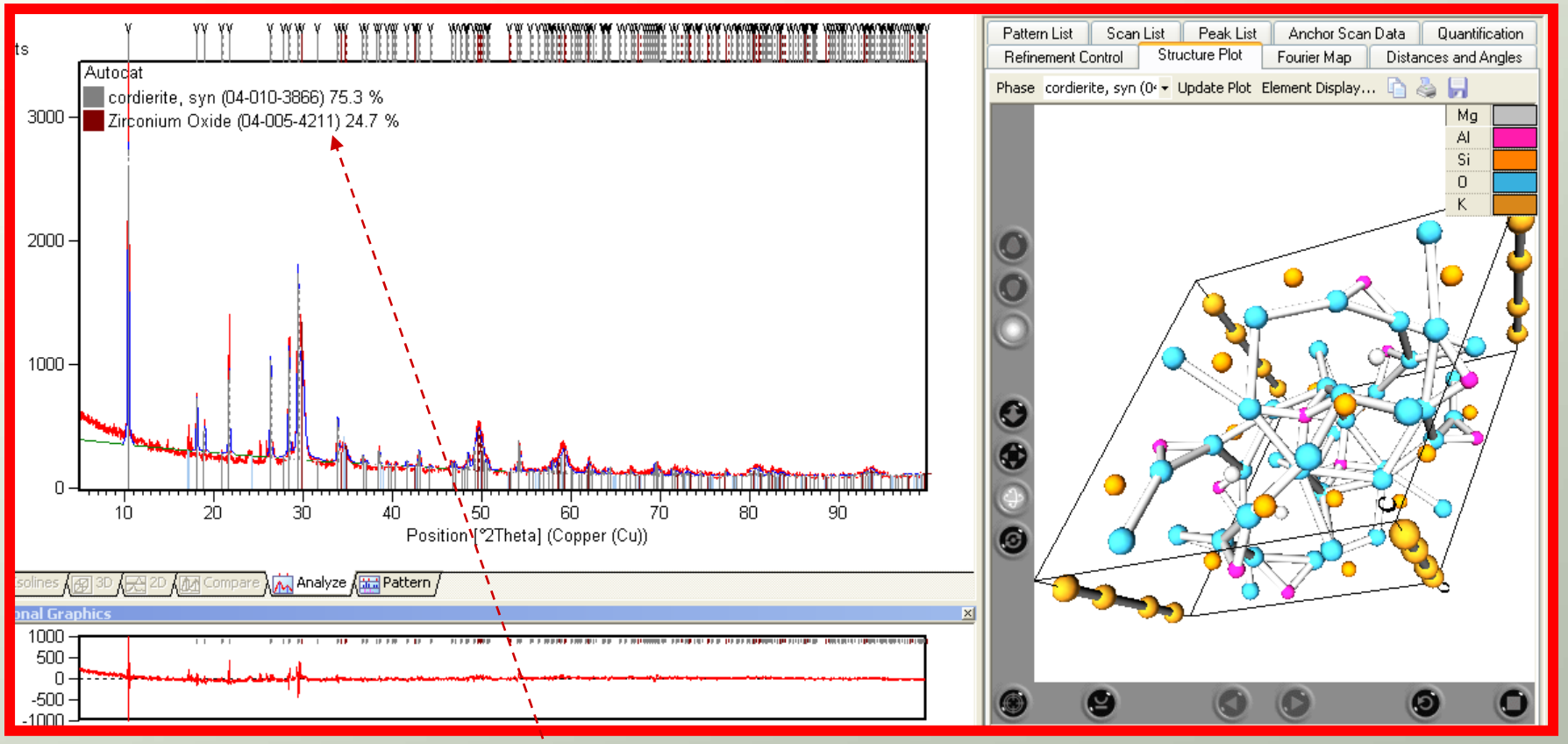
100 A

250 A

1000 A

Rietveld Analysis

This analysis used PANalytical HighScore Plus
for the Rietveld refinement



75% Cordierite, 25% Stabilized Zirconia
Experimentally Refined Cell Parameters

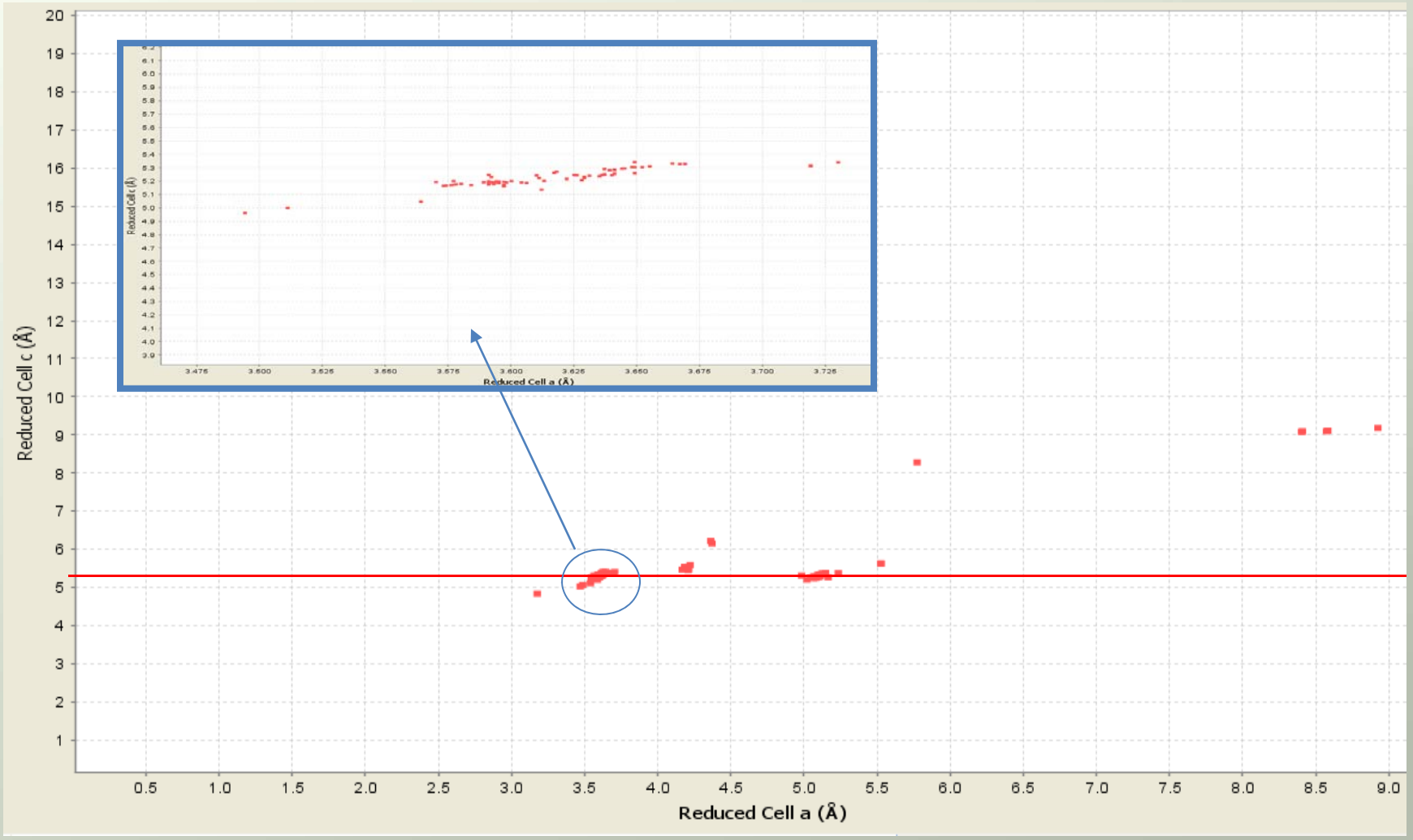
Data Mining

- Search all ZrO2 structure
- Search tetragonal space groups
- Analyze by composition, reduced cell parameters and reduced cell volume

Plot your data! (New feature in 2007!)

Point and Click!

Tetragonal Zirconia's –137 Determinations Plot of Reduced Cell a vs Reduced Cell c



Data in the PDF Entries

04-002-5421 Aug 13, 2007 3:54 PM (fawcett)

Status Primary **QM:** Prototyping (P) **Pressure/Temperature:** Ambient **Chemical Formula:** Ce0.2 Zr0.8 O2
Weight %: Ce21.07 O24.06 Zr54.87 **Atomic %:** Ce6.67 O66.67 Zr26.67
Compound Name: Cerium Zirconium Oxide

Radiation: CuKa1 **λ:** 1.5406 **d-Spacing:** Calculated **Intensity:** Calculated **I/Ic:** 10.75
Reference: "THERMAL EXPANSION OF ZIRCON REFRACTORIES". Filatov S.K., Frank Kamenetskii V.A., Zhuravina T.A. Inorg. Mater. (Engl. Transl.) 5, 286 (1969). Calculated from LPF using POWD-12++.

SYS: Tetragonal **SPGR:** P42/nmc (137) **AuthCellVol:** 69.25 **Z:** 2.00
Author's Cell [AuthCell-a: 3.637 **AuthCell-c:** 5.235 **AuthCellVol:** 69.25 **]** **Dcalc:** 6.379 **Dstruc:** 6.38
SS/FOM: F(30) = 999.9(0.000, 33) **Reference:** Ibid.

Space Group: P42/nmc (137) **Z:** 2.00 **Molecular Weight:** 133.00
Crystal Data [XtlCell-a: 3.637 **XtlCell-b:** 3.637 **XtlCell-c:** 5.235 **XtlCell.α:** 90.00 **XtlCell.β:** 90.00
XtlCell.γ: 90.00 **XtlCellVol:** 69.25 **]** **Crystal Data Axial Ratio [c/a:** 1.4394 **]**
Reduced Cell [RedCell-a: 3.637 **RedCell-b:** 3.637 **RedCell-c:** 5.235 **RedCell.α:** 90.00 **RedCell.β:** 90.00
RedCell.γ: 90.00 **RedCellVol:** 69.25 **]**

Origin: O2

Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	ITF	AET
O	1	4d	2mm.	0.25	0.25	0.05	1.0		10-a
Zr	2	2b	-4m2	0.75	0.25	0.25	0.8		8-a
Ce	3	2b	-4m2	0.75	0.25	0.25	0.2		8-a

SG Symmetry Operators:

Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator
1	x,y,z	5	x,-y+1/2,z	9	y,x,z+1/2	13	y,-x+1/2,z+1/2
2	-x,-y,-z	6	-x,y+1/2,-z	10	-y,-x,-z+1/2	14	-y,x+1/2,-z+1/2
3	-x+1/2,y,z	7	-x+1/2,-y+1/2,z	11	-y+1/2,x,z+1/2	15	-y+1/2,-x+1/2,z+1/2
4	x+1/2,-y,-z	8	x+1/2,y+1/2,-z	12	y+1/2,-x,-z+1/2	16	y+1/2,x+1/2,-z+1/2

Crystal (Symmetry Allowed): Centrosymmetric

Pearson: tP6.00 **LPF Prototype Structure:** Zr O2,tP6,137 **Subfile(s):** Inorganic, LPF Pattern, Primary Pattern
Entry Date: 01/05/2005 **Last Modification Date:** 02/03/2005

Database Comments: LPF Collection Code: 378242. Sample Preparation: STARTING MATERIALS:ZrO2. Compound Preparation: heated at 1273 K for several hours in air. Temperature of Data Collection: 293 K. Unit Cell Data Source: Powder Diffraction.

04-002-5421 (Fixed Slit Intensity) - Cu Ka1 1.54056Å

2θ	d(Å)	I	h	k	l	2θ	d(Å)	I	h	k	l	2θ	d(Å)	I	h	k	l
29.8893	**2.986900**	999	1	0	1	81.4127	1.181080	31	3	0	1	120.0131	0.889384	1	3	0	4
34.2288	2.617500	88	0	0	2	82.6594	1.166400	25	1	1	4	122.0775	0.880373	24	2	1	5
34.8571	2.571750	139	1	1	0	83.7352	1.154130	19	2	2	2	123.7751	0.873309	24	3	2	3
42.516	2.124510	11	1	0	2	84.0938	1.150120	18	3	1	0	124.6366	0.869840	24	4	1	1
49.6359	**1.834460**	299	1	1	2	88.8877	1.100070	1	3	0	2	126.1505	0.863928	20	3	1	4
50.1216	1.818500	160	2	0	0	92.961	1.062250	23	2	0	4	127.4856	0.858904	8	4	0	2
53.2832	1.717810	1	2	0	1	94.0309	1.052960	54	3	1	2	127.9359	0.857249	5	3	3	0
58.6284	1.573290	106	1	0	3	98.1269	1.019650	1	2	1	4	130.431	0.848428	1	1	0	6
59.4994	**1.553270**	201	2	1	1	99.9171	1.006140	17	1	0	5	134.2885	0.839911	1	4	1	2
62.0982	1.493450	53	2	0	2	101.3671	0.995633	16	3	0	3	137.5816	0.826245	9	1	1	6
67.775	1.381510	3	2	1	2	102.0948	0.990502	32	3	2	1	141.9972	0.814671	15	3	3	2
72.11	1.308750	14	0	0	4	109.8423	0.941246	1	3	2	2	142.5796	0.813258	16	4	2	0
73.6015	1.285870	34	2	2	0	114.2356	0.917231	14	2	2	4	149.2086	0.798950	1	3	2	4
77.4387	1.231450	2	1	0	4	115.8082	0.909250	9	4	0	0						
80.6922	1.189800	64	2	1	3	116.1903	0.907357	5	2	0	5						

Author, Crystal Reduced Cells

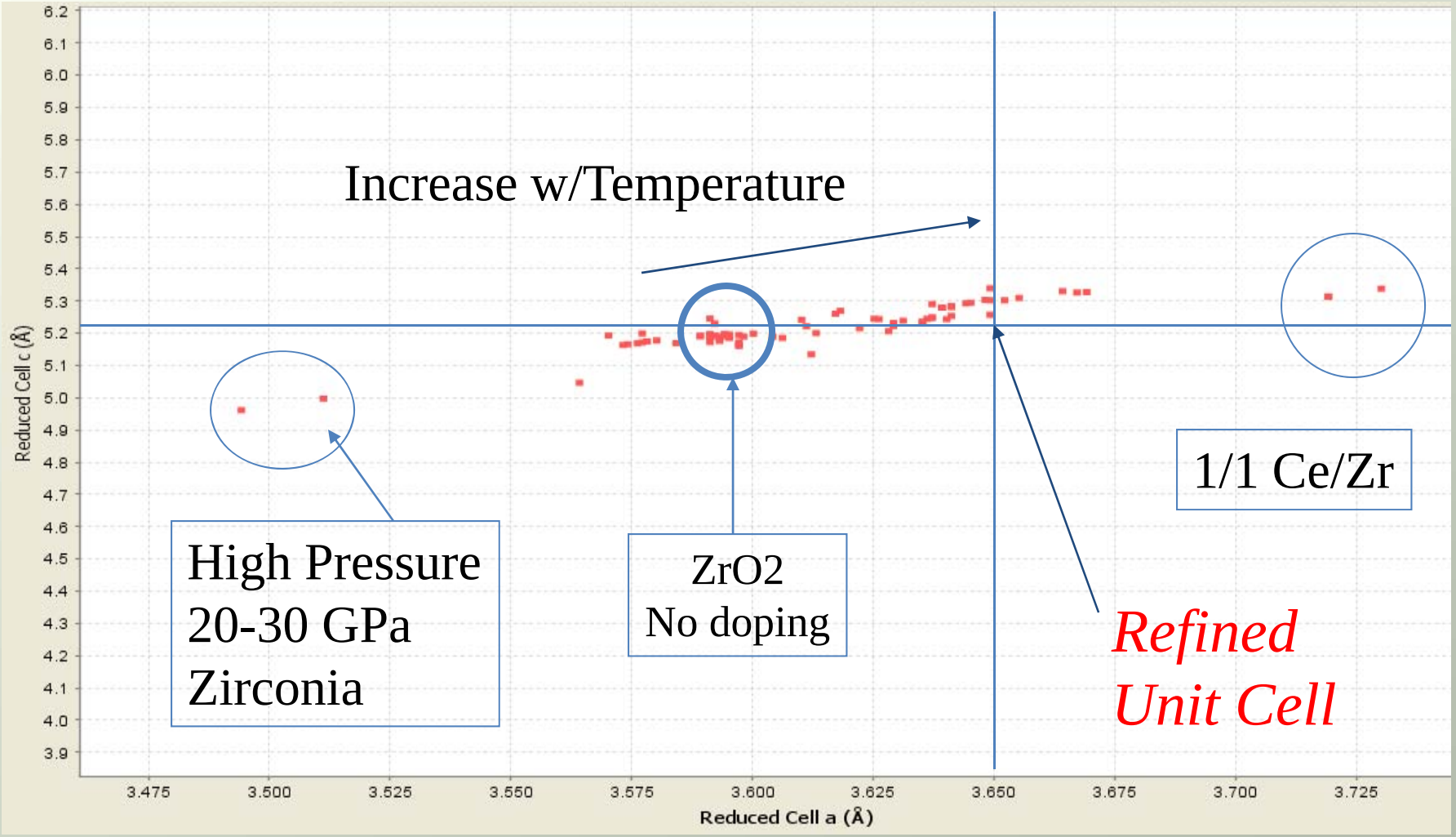
Atomic Parameters and symmetry

Editor's comments

Indexed pattern

Use editors comments to interpret the data!

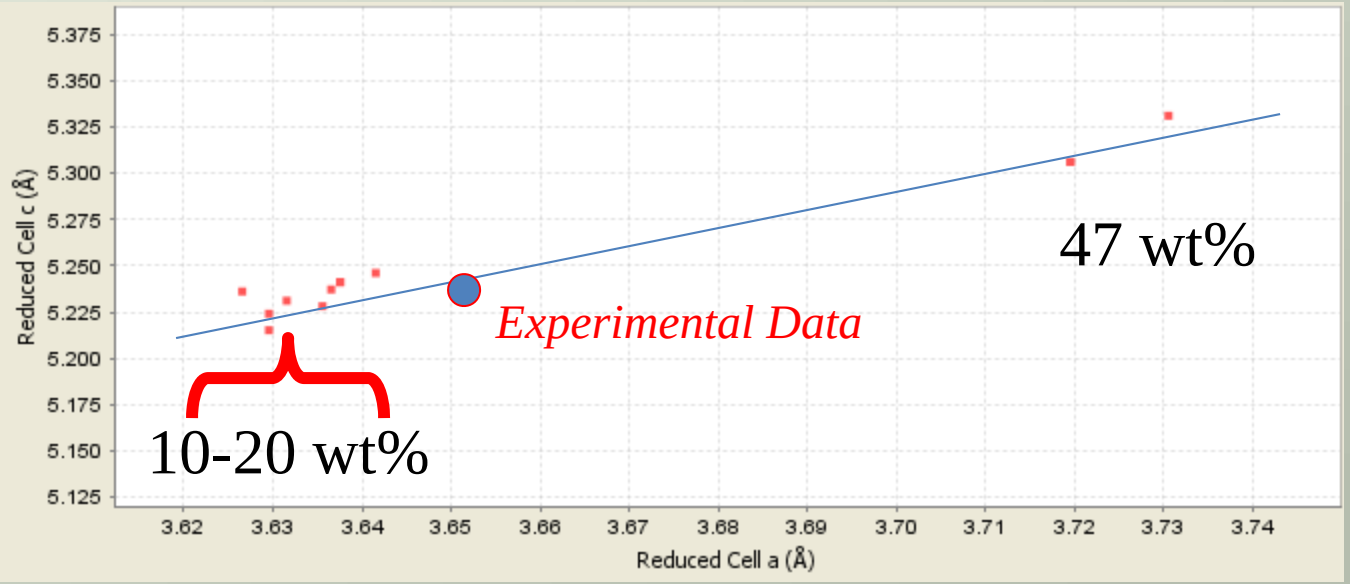
Best matches with cell dimensions
- Ce Stabilized Zirconia
- ZrO2 made at 1200 K



Ce doped Zirconia

10 Determinations

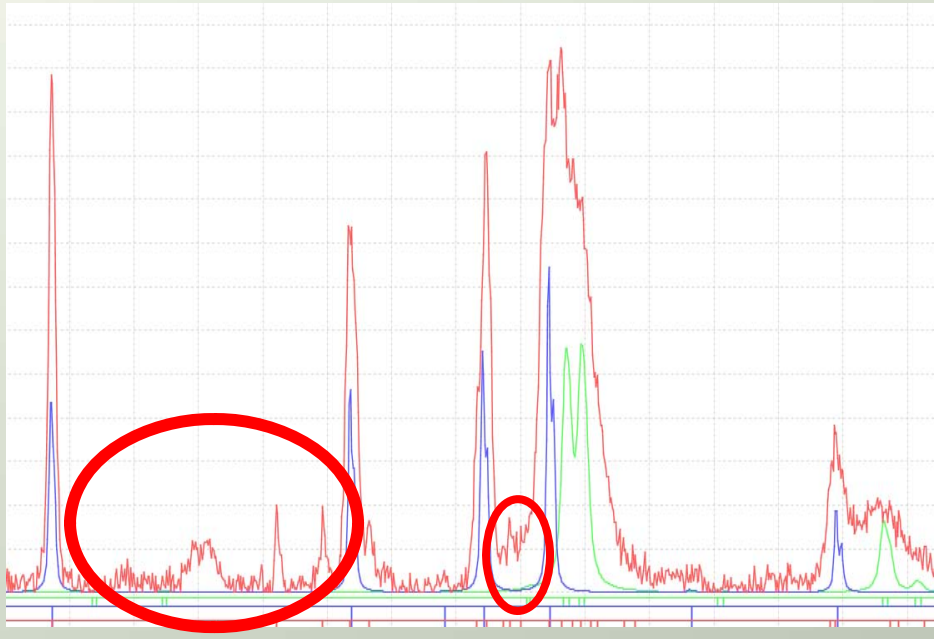
PDF #	QM	Chemical Formula	Compound Name	Weight % ▲	Atomic %	SYS	RedCell-a	RedCell-c	RedCellVol
04-006-7959	S	Ce0.1 Zr0.9 O2	Cerium Zirconium Oxide	Ce10.94 O24.98 Zr64.08	Ce3.33 O66.67 Zr30.00	T	3.630	5.213	68.68
01-082-1398	S	(Zr0.88 Ce0.12) O2	Zirconium Cerium Oxide	Ce13.03 O24.79 Zr62.19	Ce4.00 O66.67 Zr29.33	T	3.627	5.234	68.85
01-089-9067	S	(Zr0.88 Ce0.12) O2	Zirconium Cerium Oxide	Ce13.03 O24.79 Zr62.19	Ce4.00 O66.67 Zr29.33	T	3.636	5.226	69.09
04-006-7961	I	Ce0.12 Zr0.88 O2	Cerium Zirconium Oxide	Ce13.03 O24.79 Zr62.19	Ce4.00 O66.67 Zr29.33	T	3.630	5.222	68.79
04-006-7962	I	Ce0.15 Zr0.85 O2	Cerium Zirconium Oxide	Ce16.10 O24.51 Zr59.39	Ce5.00 O66.67 Zr28.33	T	3.632	5.229	68.99
00-038-1437	I	Zr0.84 Ce0.16 O2	Cerium Zirconium Oxide	Ce17.11 O24.42 Zr58.47	Ce5.33 O66.67 Zr28.00	T	3.638	5.239	69.33
04-007-4922	I	Ce0.16 Zr0.84 O2	Cerium Zirconium Oxide	Ce17.11 O24.42 Zr58.47	Ce5.33 O66.67 Zr28.00	T	3.638	5.239	69.33
01-080-0785	S	Zr0.82 Ce0.18 O2	Zirconium Cerium Oxide	Ce19.10 O24.24 Zr56.66	Ce6.00 O66.67 Zr27.33	T	3.642	5.244	69.56
04-002-5421	P	Ce0.2 Zr0.8 O2	Cerium Zirconium Oxide	Ce21.07 O24.06 Zr54.87	Ce6.67 O66.67 Zr26.67	T	3.637	5.235	69.25
00-038-1436	I	Zr0.5 Ce0.5 O2	Cerium Zirconium Oxide	Ce47.44 O21.67 Zr30.89	Ce16.67 O66.67 Zr16.67	T	3.720	5.304	73.42
00-055-0997	I	Ce0.5 Zr0.5 O2	Cerium Zirconium Oxide	Ce47.44 O21.67 Zr30.89	Ce16.67 O66.67 Zr16.67	T	3.731	5.329	74.19
04-002-2715	P	Ce0.5 Zr0.5 O2	Cerium Zirconium Oxide	Ce47.44 O21.67 Zr30.89	Ce16.67 O66.67 Zr16.67	T	5.260	5.300	146.64
04-007-4923	I	Ce0.5 Zr0.5 O2	Cerium Zirconium Oxide	Ce47.44 O21.67 Zr30.89	Ce16.67 O66.67 Zr16.67	T	3.720	5.304	73.42



Cordiertie ZrO2

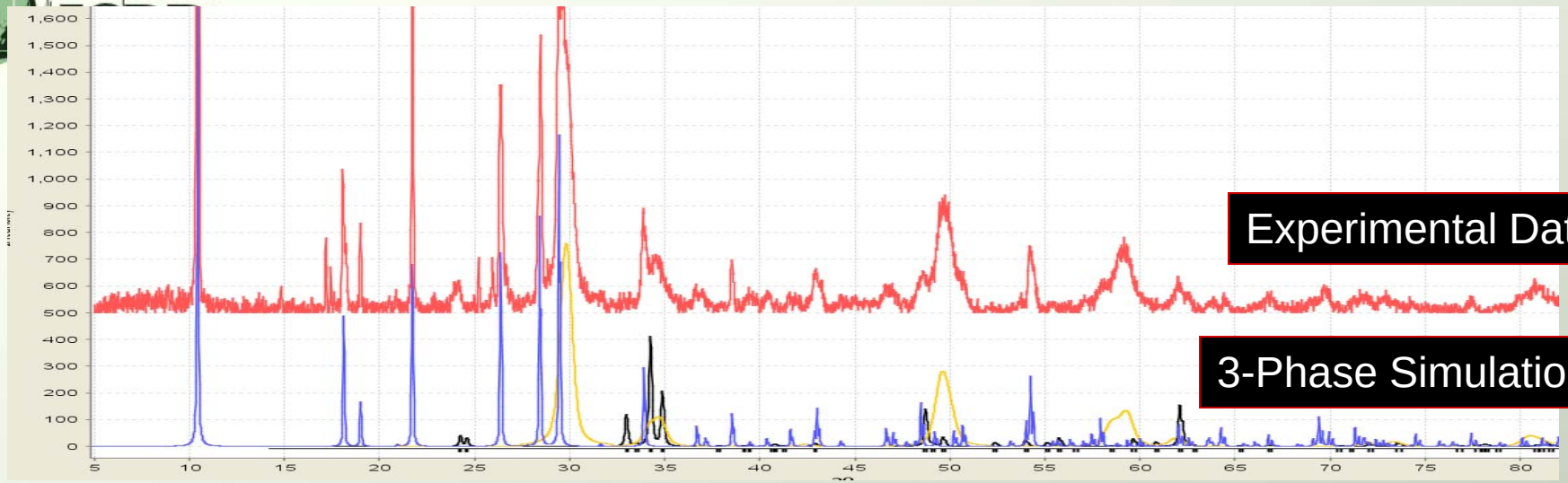
Lines (30 of 59)

Ex d(Å) ▼	Ex I	P1 d(Å)	P1 I	P2 d(Å)	P2 I
11.30794	2				
8.44311	100	8.481510	100		
5.16004	9				
4.90437	15	4.896800	18		
4.85981	8			4.823000	5
4.66904	11	4.669250	7		
4.08603	38	4.090370	27		
3.71682	3				
3.66636	2				
3.52993	6				
3.43370	6				
3.38019	27	3.379240	28		
3.34271	5				
3.14912	14				
3.13282	33	3.139250	32		
3.10428	3				
3.09371	5				
3.07626	5			3.067000	1
3.03184	40	3.032030	43		
3.01178	41			3.008000	100
2.99527	33				
2.98220	29			2.983000	100
2.96763	22				
2.95800	16				
2.91857	3				



Additional Small Phases

Requires
- Better Data or
- Specimen History or
- Elemental Analysis



Deduction

~3% RhO2 (black pattern in above simulation)
on a Ce doped tetragonally stabilized ZrO2 washcoat
used with a cordierite honeycomb substrate

Substrate is large crystallite size, washcoat is small crystallite size

Consistent with XRD data, but also consistent with patent literature researched on the Internet

World Intellectual Property Organization
Result of an Internet Search

A supported catalyst useful in the present invention was prepared as follows:

Onto a 400 cells/in2 (62 cells/cm2) cordeirite honeycomb monolith, is deposited a catalyst washcoat underlayer of a slurry of a mixture of alumina, ceria and zirconia to give a total deposit of 2. Og/in3 (0.12g/cm3).
The resulting monolith is fired for 1 hour in air at 500°C. A first catalyst layer is deposited onto the monolith by impregnating the washcoated monolith with a mixed solution of tetramine platinum dichloride and barium acetate, to yield an intimate mixture of platinum and barium.
The barium acetate is deposited at a loading of 800g/ft3 of Ba (28g/litre) and the platinum is deposited at a loading of 100g/ft 3of Pt (3.5g/litre).
The monolith is fired again under the same conditions. A second washcoat layer is then deposited to yield a deposit of 1. Og/in3 (0.06g/cm3) of ceria-stabilized zirconia (11% Ce02,89% ZrO2) in admixture with a solution of rhodium nitrate, to yield a deposit of 6g/ft3 (0.21 g/litre) of Rh in the second washcoat layer.
The treated monolith is fired again under the same conditions and then a second impregnation is carried out using cerium acetate solution to deposit 400g/ft3 (14g/litre) of Ce. The monolith is fired again under the same conditions

Conclusions

- The XRD pattern directly yields the identification of cordierite and zirconia and their respective crystallite sizes from an analysis of peak locations and profiles.
- Rietveld refinement provides a quantitative analysis and refined cell parameters which results in the identification of Ce doping in the zirconia unit cell.
- Data mining results in a concentration for the zirconia doping and a synthesis temperature for the cordierite and zirconia by references to the known literature data in PDF-4+.
- The above data can be used on a search on the internet, which identifies catalytic converters having these components
- Converter patents indicate that Rh, Pt and Pd are likely catalysts.

  RhO_2 does fit as a minor (~3%) phase, other oxides are possible but are heavily overlapped with the zirconia pattern.

Based on the above, additional analyses should be able to confirm the synthesis route and other phases present in the specimen.

Thank you for viewing our tutorial.

Additional tutorials are available at the ICDD web site (www.icdd.com).

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