

# **Powder X-ray Diffraction Profile Analysis Software PDXL Introduction**

## PDXL 2.7

- System requirements: Windows 7 Pro 32-bit / 64-bit or 8.1 Pro 64-bit
- Windows 10 is officially not yet supported
- Needs the Microsoft C++ Redistributive 2008 package incl. updates and an USB dongle driver (Sentinel)
- The latest PDXL version can be obtained from <http://www.rigaku.com/en/service/software/pdxl>
- English format for numbers to separate digits is required (e.g. 0.3 *not* 0,3)!
- Recommended freeware tool for data conversion, e.g. to import measured data from other systems: “PowDLL” <http://users.uoi.gr/nkourkou/powdll/>
- Get the free crystallographic database (COD) with >350k entries: <http://www.crystallography.net>
- Demo data for testing is available under C:\Users\Public\Documents\Rigaku\PDXL2\DemoData
- See also the help files available in pdf-format!

# TOPICS for PDXL

- Function and analysis example with X-ray diffraction multifunctional software PDXL
  - Qualitative analysis
  - Quantitative analysis
  - Application
  - WPPF method and Rietveld analysis

# Understand from X-ray Diffraction

Peak position

lattice spacing → Qualitative  
Lattice parameter

d value shift → Residual stress  
Solid solution analysis

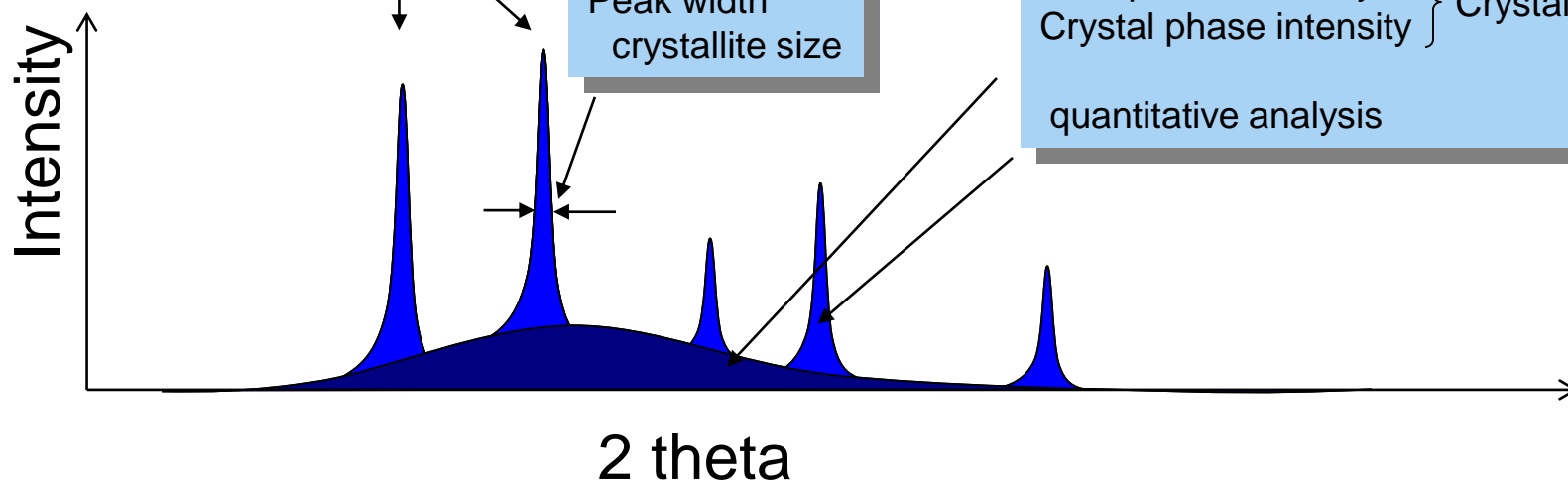
sample direction & peak intensity ratio  
(preferred orientation)

Texture  
fiber structure  
pole figure

Peak width  
crystallite size

Amorphous intensity  
Crystal phase intensity } Crystallinity

quantitative analysis



# Analytical Technique of X-ray Diffractometry

Qualitative analysis

Quantitative analysis

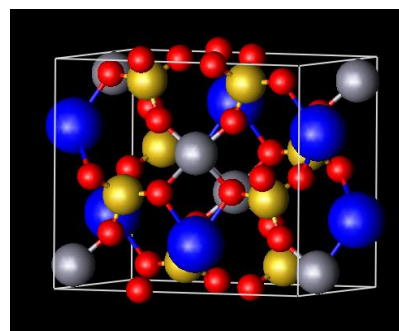
Crystallinity

Lattice parameter refinement

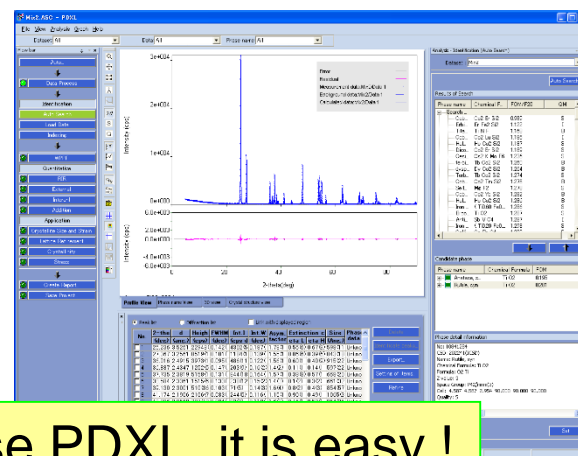
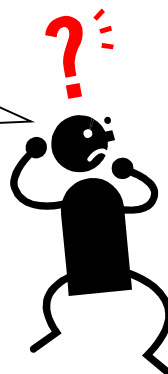
Crystallite size & lattice strain

Rietveld analysis

No.	h	k	l	$2\theta (\lambda=1)$	I (相对)
1	1	0	1	25.3066	100.0
2	0	1	-3	36.9504	6.6
3	0	0	4	37.7916	20.7
4	1	1	-2	38.5700	7.6
5	0	2	0	48.0430	29.6
6	-1	0	5	53.8855	19.2
7	1	2	-1	55.0679	18.9
8	-2	-1	-3	62.1131	3.3
9	0	2	-4	62.6892	15.1
10	-1	1	6	68.7562	6.8
11	-2	2	0	70.2970	7.6
12	-1	0	-7	74.0518	0.6
13	-2	-1	-5	75.0502	11.9



I want to analyze it but it seems difficult



If you use PDXL, it is easy !

# PDXL: Integrated Analytical Software for Powder X-ray Diffraction

- The qualitative analysis is improved by the hybrid search/match.
  - The combination of peak search and profile fitting is a unique technology for qualitative analysis.
- Everyone can do the quantitative analysis by using Rietveld method.
  - Due to interface all users from beginners to experts can easily do quantitative phase analysis by Rietveld method.
- The operation time can be reduced significantly by the package analysis with the automatic function.
  - A lot of data can be analyzed automatically under the same analytical content and an analytical conditions.
  - The report creation and the preservation of analytical results are automatically done.

# Integrated Analytical Software for Powder X-ray Diffraction Data 'PDXL'

The screenshot displays the PDXL software interface with several key components highlighted by colored boxes and labels:

- Flow bar:** A vertical toolbar on the left side containing buttons for 'Auto...', 'Data Process', 'Identification', 'Auto Search', 'Load Data', 'Indexing', 'WPPF', 'Quantitative', 'RIR', 'External', 'Lattice Refinement', 'Crystallinity', 'Stress', 'Create Report...', and 'Save Project...'.
- Indication area:** A central plot showing Intensity (cps) versus 2-theta (deg). The plot features a sharp peak at approximately 25.2 degrees 2-theta, with a yellow label 'Indication area' pointing to it.
- Analysis area:** A window on the right titled 'Analysis - Identification (Auto Search)' showing the 'Results of Search' table and 'Candidate phase' table.
- Information area:** A table at the bottom showing a list of peaks with columns for No., 2-theta (deg), d (Ang.), Height (cps), FWHM (deg), Int.I (cps/d), Int.W (deg), Asym. factor, Extinction c, Size (Ang.), and Phase data.

**Results of Search Table:**

Phase name	Chemical F...	FOM/F20	QM
Search ...			
Cob...	Co2 Er Si2	1.089	S
ErbL...	Er Fe2 Si2	1.191	I
Tita...	Ti N F	1.245	I
Cob...	Co2 Lu Si2	1.250	I
Cesi...	Ce2 K Mo F6	1.257	S
Hol...	Ho Co2 Si2	1.258	S
Dico...	Co2 Er Si2	1.258	S
ALP...	LiI O3	1.262	C
Mag...	Mg F2	1.290	S
Iron ...	(Ti0.60 Fe0...	1.292	S
Broo...	Ti O2	1.299	S
CHR...	CR SB O4	1.301	S
Iron ...	(Ti0.20 Fe0...	1.303	S
Galli...	Ga Sb O4	1.304	S

**Candidate phase Table:**

Phase name	Chemical Formula	FOM
TITANIUM ...	Ti O2	0.123
TITANIUM ...	Ti O2	0.109

**Peak list Table:**

No.	2-theta (deg)	d (Ang.)	Height (cps)	FWHM (deg)	Int.I (cps/d)	Int.W (deg)	Asym. factor	Extinction c	Size (Ang.)	Phase data
1	25.236	3.5261	22948	0.142	4302.5	0.187	1.78	0.56	0.67	598.11
2	27.367	3.2561	8519	0.101	1184.0	0.139	1.55	0.86	0.39	843.18
3	36.016	2.4915	3973	0.095	486.0	0.122	1.56	0.60	0.40	915.22
4	36.887	2.4347	1252	0.147						
5	37.735	2.3819	5158	0.131						
6	38.504	2.3361	1515	0.133						
7	39.130	2.3001	510	0.103						
8	41.174	2.1906	2106	0.083						
9	43.986	2.0569	707	0.094						
10	47.975	1.8947	7513	0.124	1246.0	0.166	1.32	0.59	0.67	730.0
11	53.832	1.7015	4709	0.125	743.0	0.158	1.18	0.60	0.35	746.0
12	54.266	1.6890	5364	0.0954	649.0	0.121	1.38	0.60	0.35	977.0
13	55.002	1.6691	1462	0.120	730.0	0.164	1.16	0.50	0.60	477.0

# New Finding From Many Data Analysis

**A lot of conditions**

temperature  
humidity  
pressure  
combination

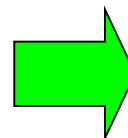
▪  
▪  
▪

**VS**

lattice parameter  
crystallite size  
crystallinity  
radial stress  
crystallography  
quantitative value



**From many data in many conditions  
A material finding is easily acquired in one batch.**

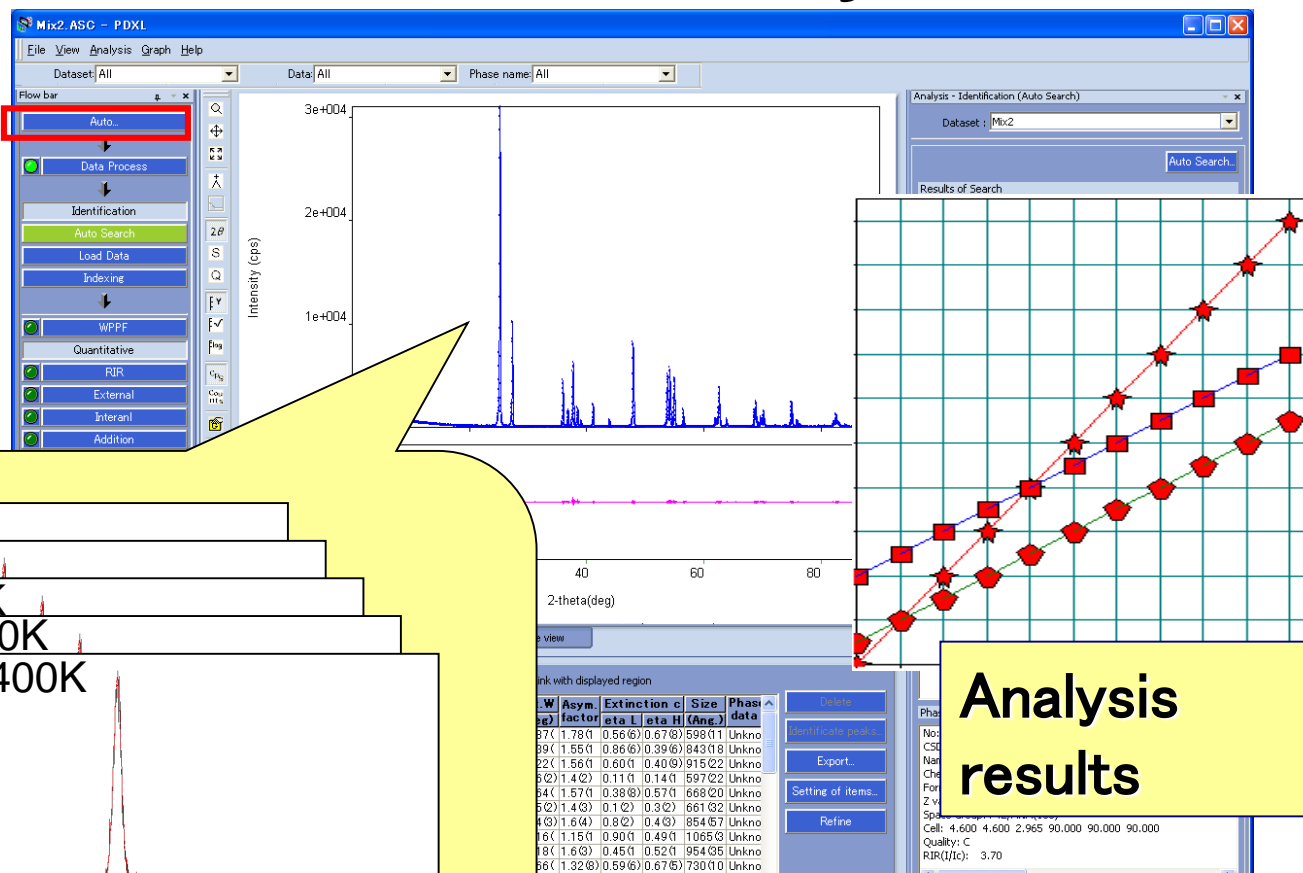


**Application to material  
development**

**For example: The comparison of crystal structure information  
according to the temperature change etc. are effective.**



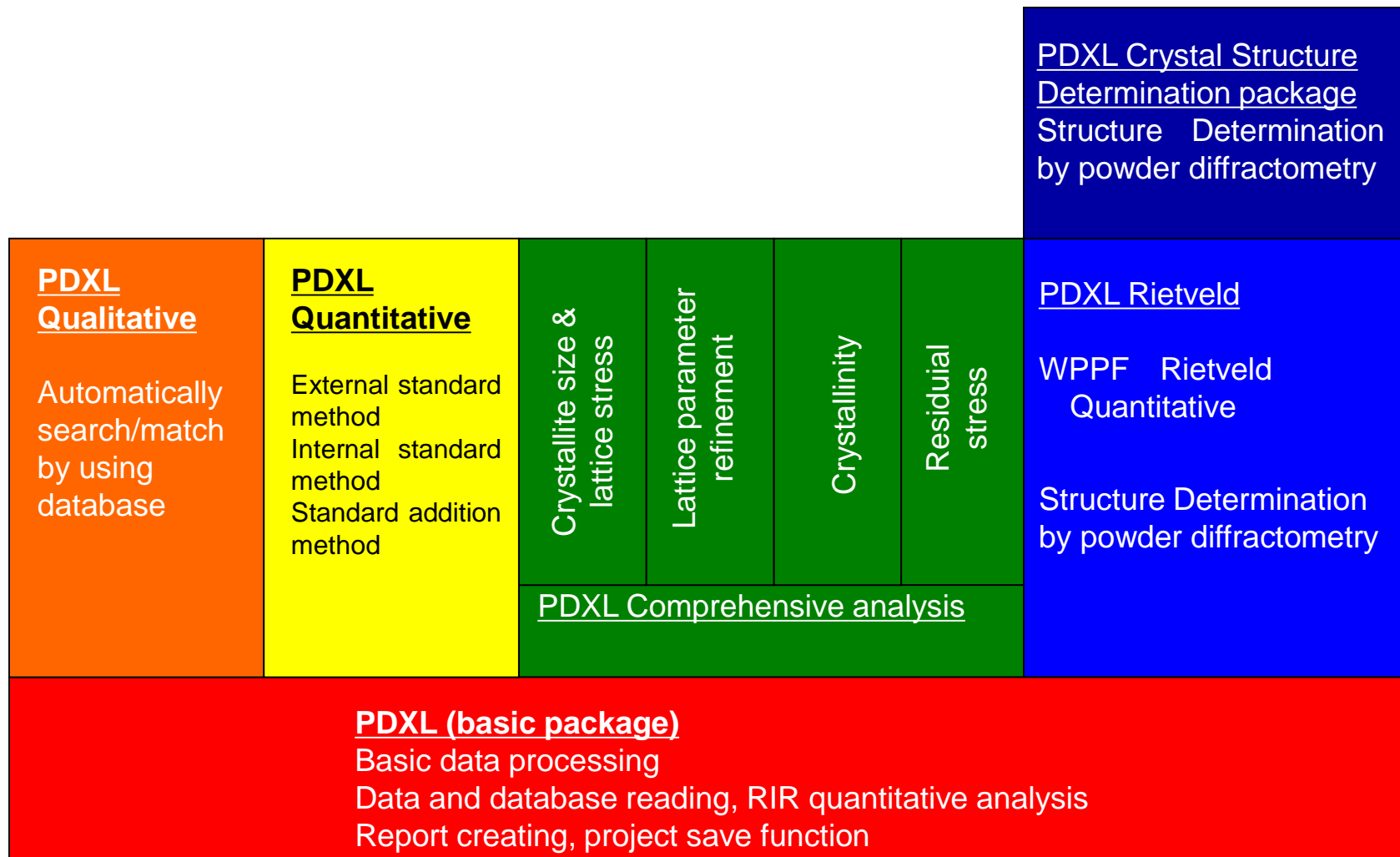
# A lot of Data Analysis



one batch analysis of a lot of data with different temperatures

Relation between temperature and analytical result

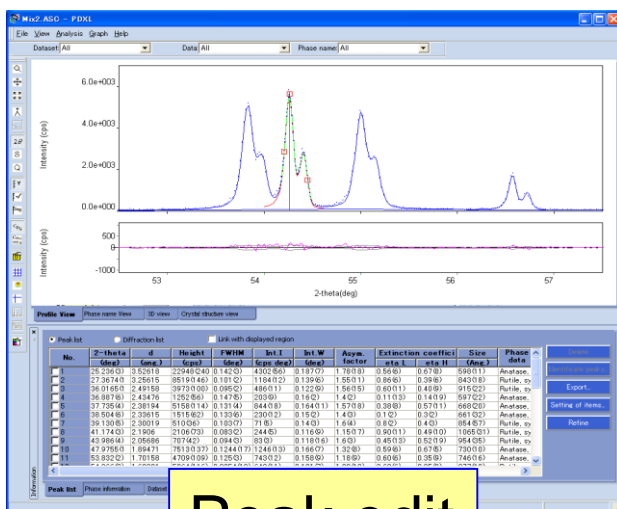
# Structure of PDXL



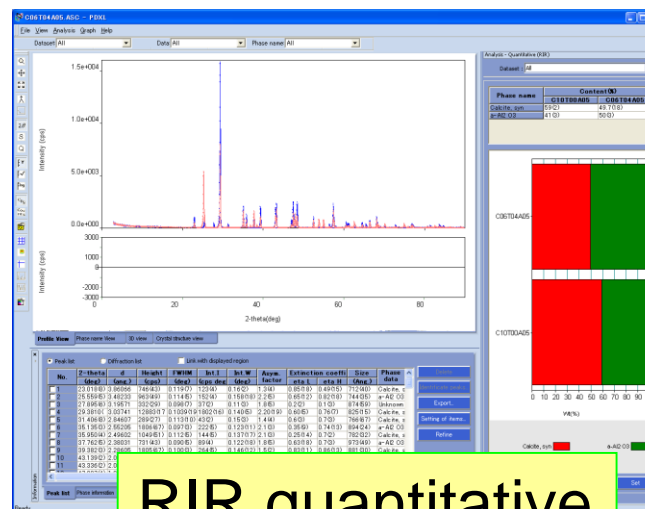
# PDXL Basic Package

## • Basic data processing

- The automatic profile fitting is executed by data reading.
- Detailed information can be obtained on the peak, such as peak position, peak width, integrated intensity, and crystallite size (by Scherrer method) etc.
- RIR (relative intensity ratio) quantitative analysis is possible.



Peak edit

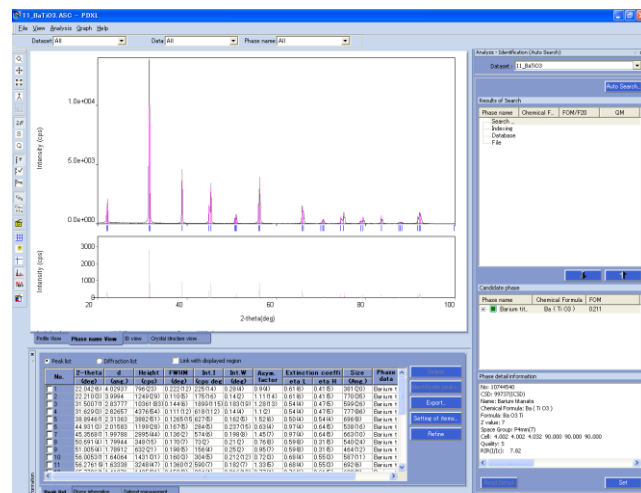


RIR quantitative

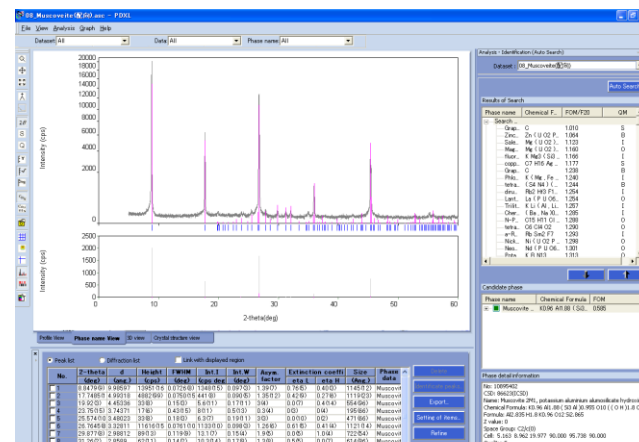
# PDXL Qualitative

## • Hybrid search match

- Peak search combined with profile fitting provide more flexibility
- PDXL supports different diffraction databases, such as ICDD PDF-2, PDF-4+, COD and ICSD
- Crystallographic Information files '.cif' can be imported
- User database can be created.



Detection of lattice transformation



The presence of preferred orientation can be judged

## ICDD PDF-4+

The PDF-4+ is divided into groups:

**00-** experimental data, **01-** ICSD cards, **02-** CSD (“PDF-4 organics”),  
**03-** NIST metals & alloys, **04-** cards with atom coordinates

## User database

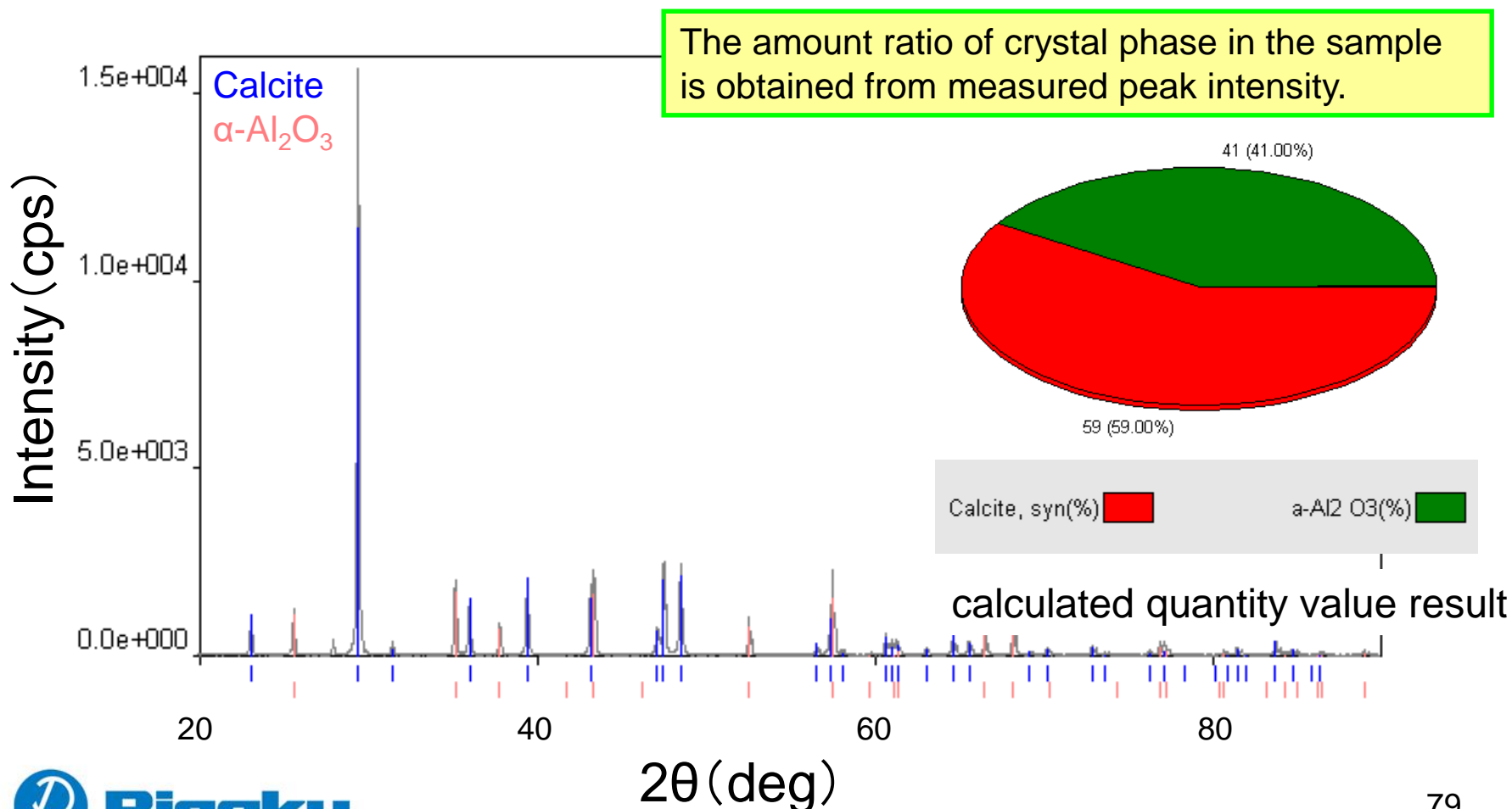
For creation of a user database see PDXL user manual chapter 1.3.4  
In order to copy or remove an existing user database go to the following directory and copy or remove all three files with the same name but different extensions (.db, .db.idx, .db.seek):

C:\ProgramData\Rigaku\Database\PDXL2\USER

Attention: ProgramData is a hidden directory, so first activate displaying all files

# Quantitative Analysis

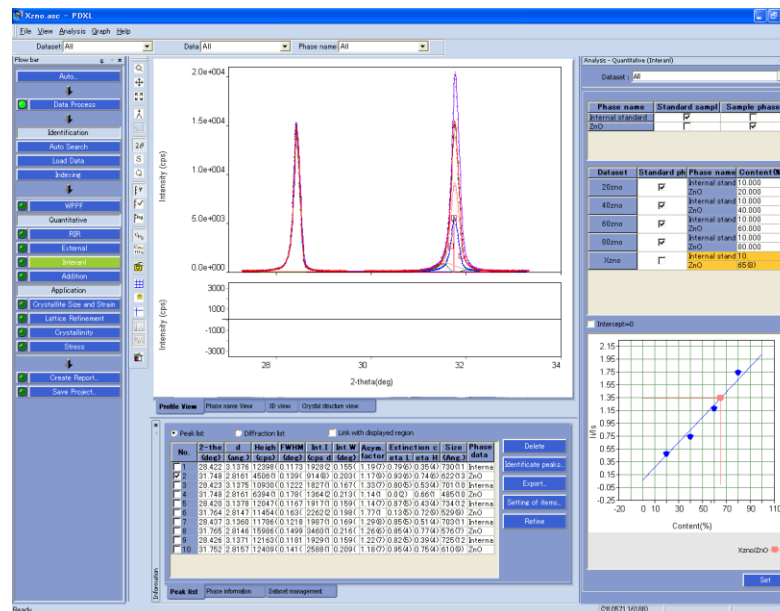
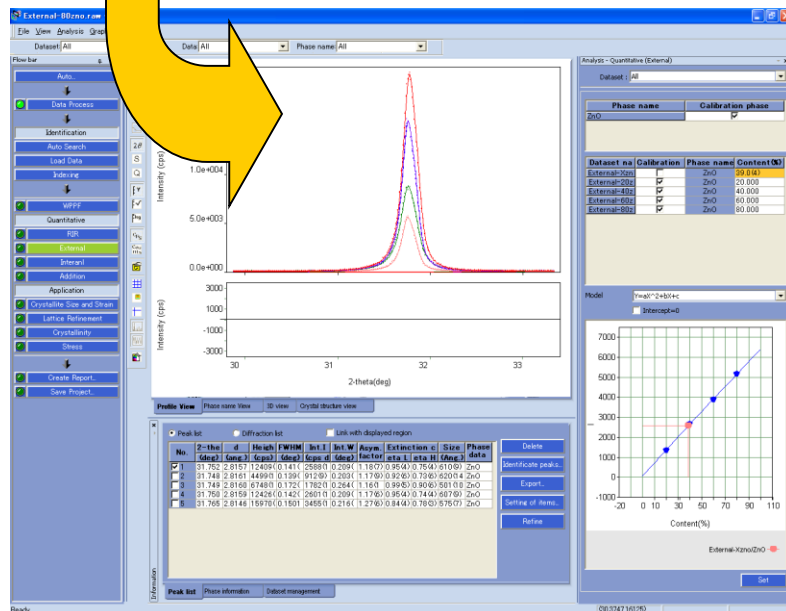
- How much of each phase is there?



# PDXL: Quantitative Analysis

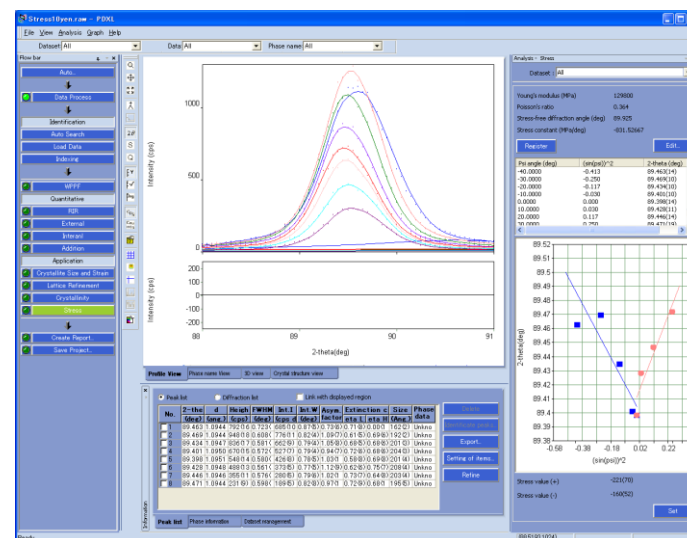
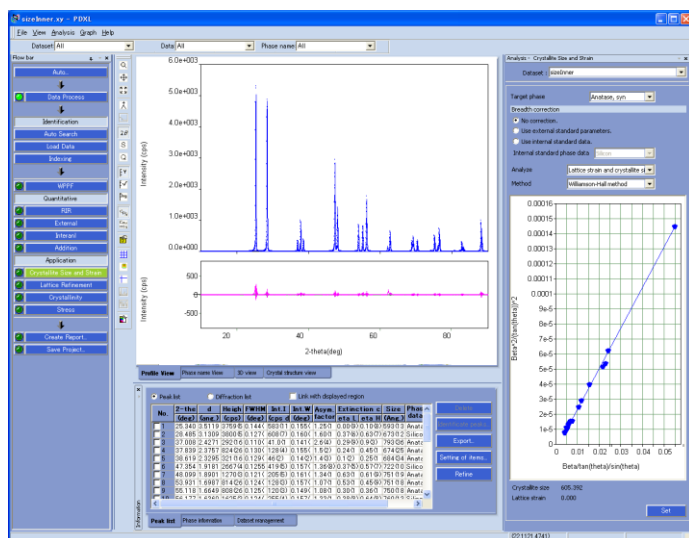
- External standard method, Internal standard method, Standard addition method
- The quantitative analysis of a specific phase by the calibration curve method is possible, easy and prompt.

It is convenient for the process control etc.



# PDXL: Application Analysis

- Crystallite size, Lattice strain, Lattice parameter refinement, Crystallinity, Residual stress, Indexing, Structure determination
  - Various applications and analysis results are immediately obtained according to an accurate peak position, width and the integrated intensity obtained by the peak deconvolution during the profile fitting.
  - It is effective for the comparison of similar samples and to evaluate physical properties indirectly.





# Crystallite size & Lattice strain

Crystallite size can be estimated in a very easy way by using Scherrer equation (included in PDXL basic):

$$B(2\theta) = \frac{0.94\lambda}{L \cos(\theta)}$$

where  $L = \sqrt{L_{obs}^2 - L_{ins}^2}$ , L in radians.

A **combined size-strain analysis** is possible due to angle-dependency of peak broadening:

- Size broadening: all peaks are broad,
- Strain broadening: low angle peaks are significantly sharper

**Strain-broadening:**  $B(2\theta) = 4\varepsilon_0 \tan \theta$ ,  $\varepsilon_0 = \frac{\Delta d}{d}$

Lattice strain and crystallite size can be calculated by using **Halder-Wagner method** and **Williamson-Hall plot** (implemented in PDXL).