# 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 Diffractometry

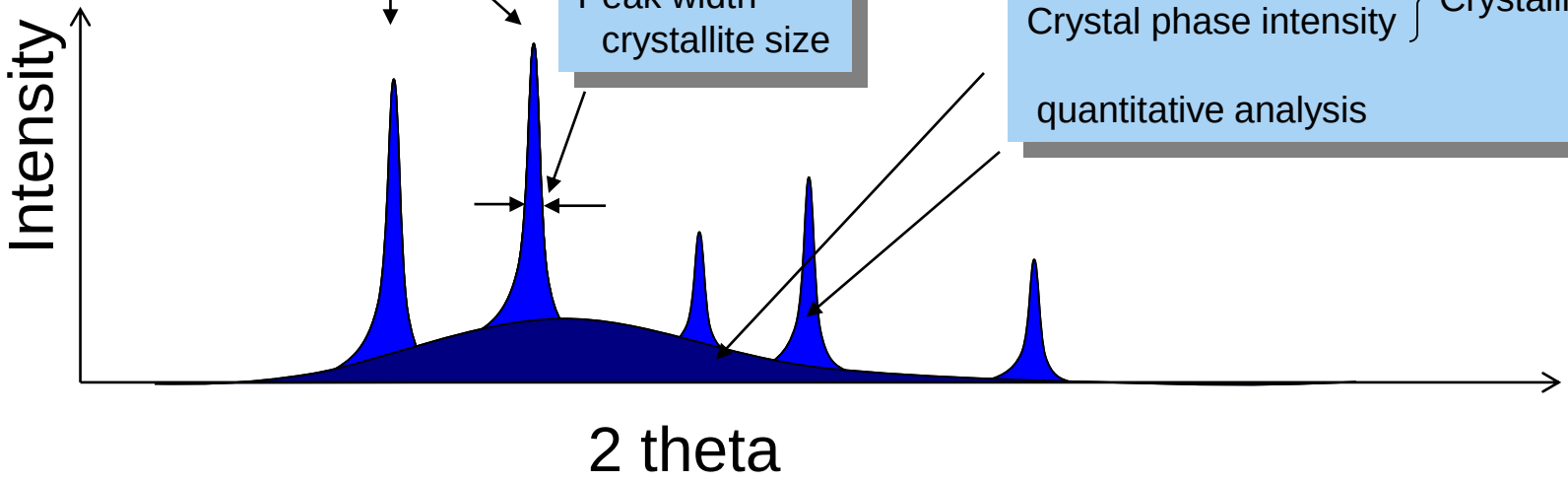
Peak position
lattice spacing → Qualitative
Lattice parameter
d value sift → Residual stress
Solid solution analysis

sample direction & peak intensity ratio
(preferred orientation)
Texture
fiber structure
pole figure

Peak width
crystallite size

Amorphous intensity
Crystal phase intensity
} Crystallinity

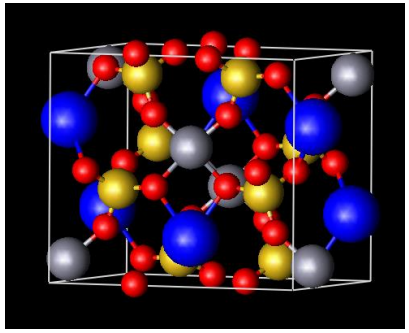
quantitative analysis

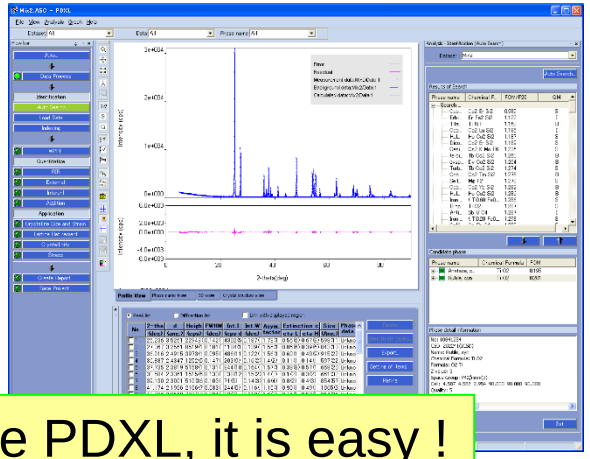


# Analytical Technique of X-ray Diffractometry

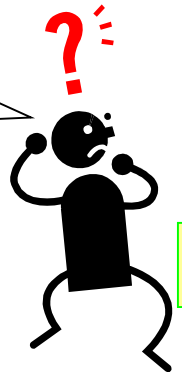
Qualitative analysis
Quantitative analysis
Crystallinity
Lattice parameter refinement
Crystallite size & lattice strain
Rietveld analysis

| No. | h | k | l | 2θ(λ=1. | I(相対 |
|---|---|---|---|---|---|
| 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

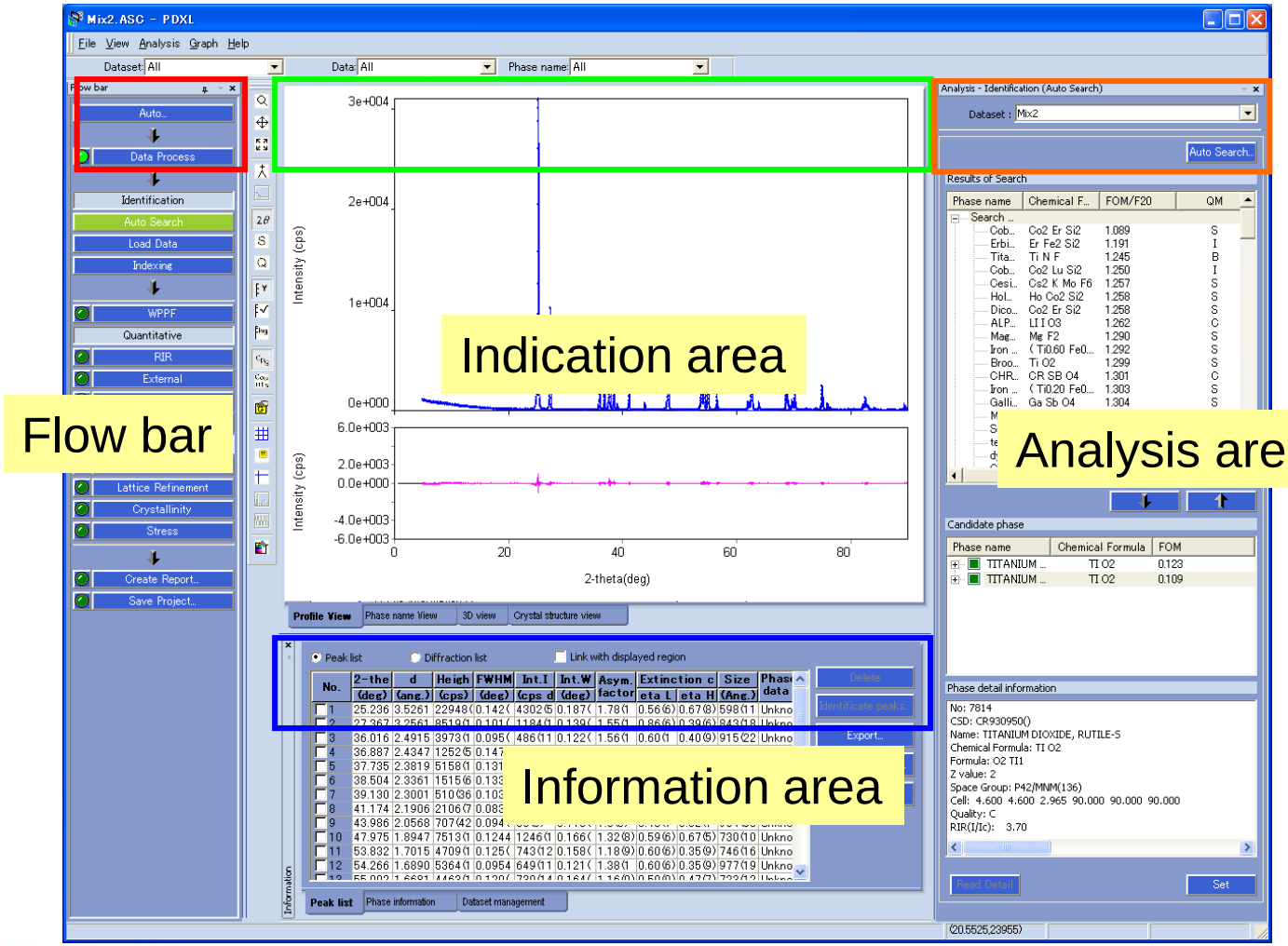


If you use PDXL, it is easy !

# 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'



# New Finding From Many Data Analysis

**A lot of conditions**

**temperature**
**humidity**
**pressure**
**combination**
**.**
**.**
**.**

**VS**

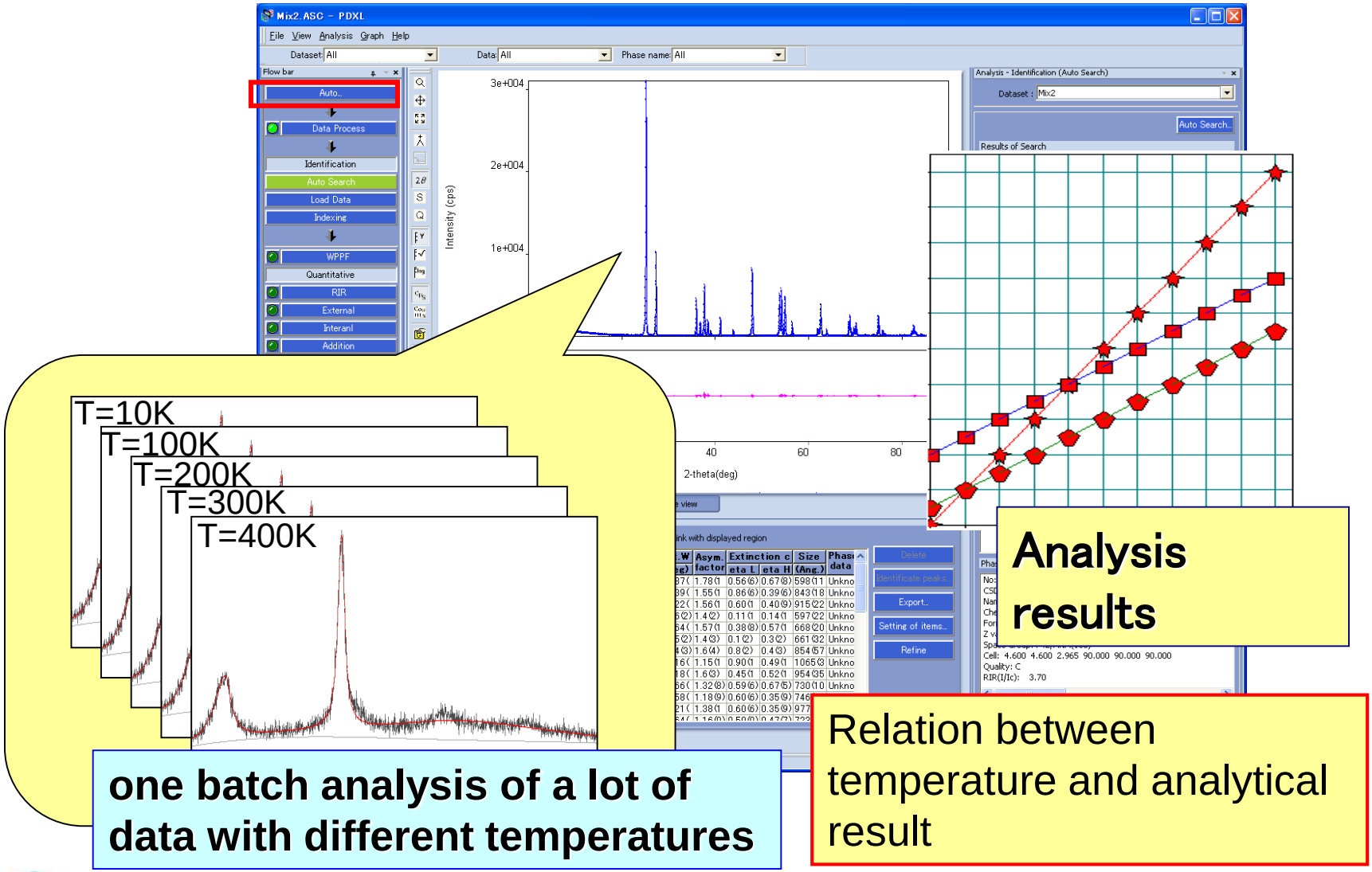
**lattice parameter**
**crystallite size**
**crystallinity**
**radial stress**
**crystallography**
**quantitative value**

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

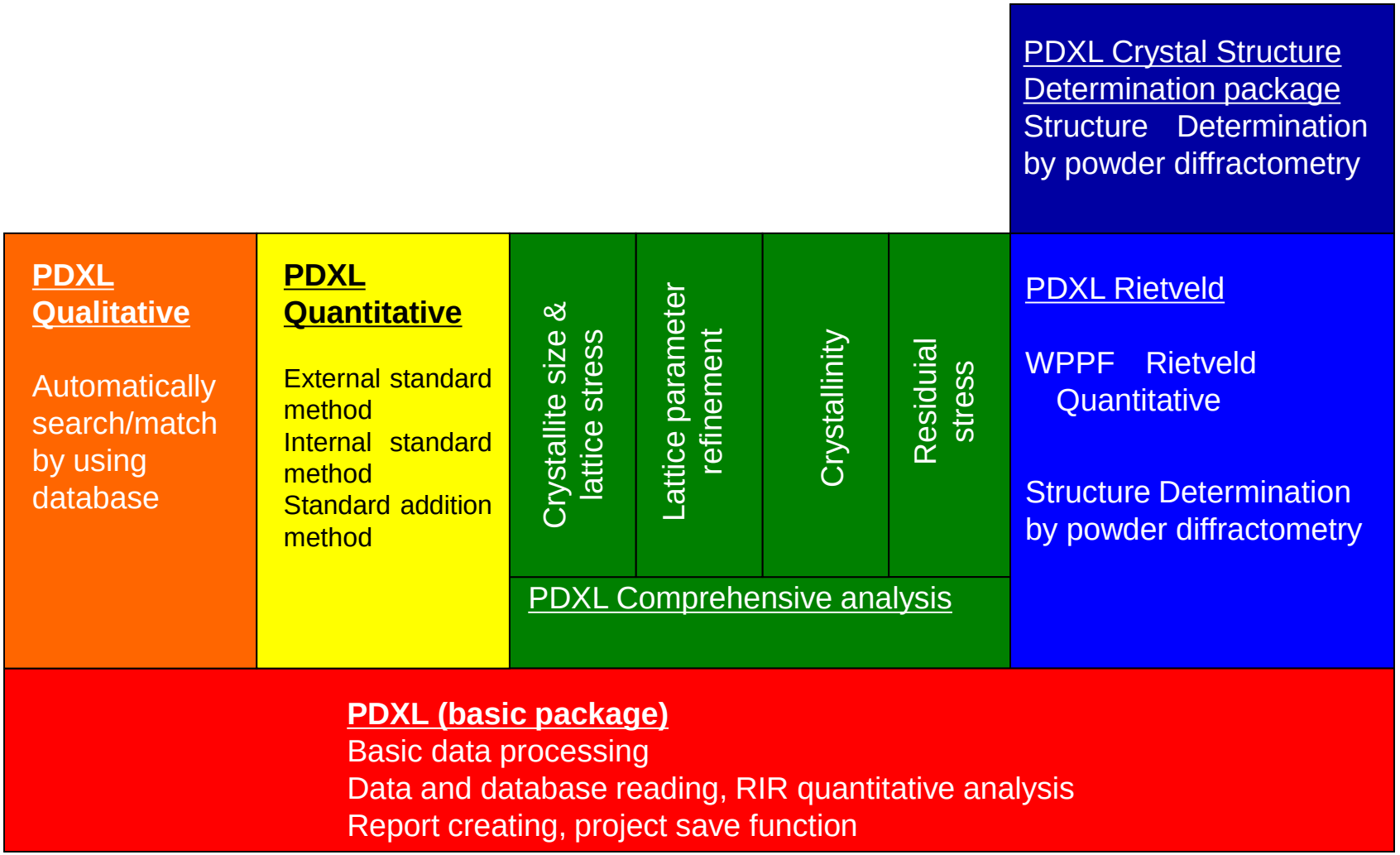
**Application to material development**

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

# A lot of Data Analysis

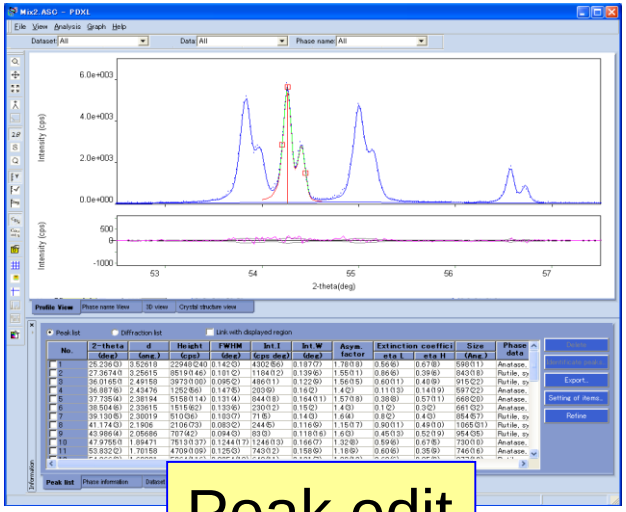


# Structure of PDXL

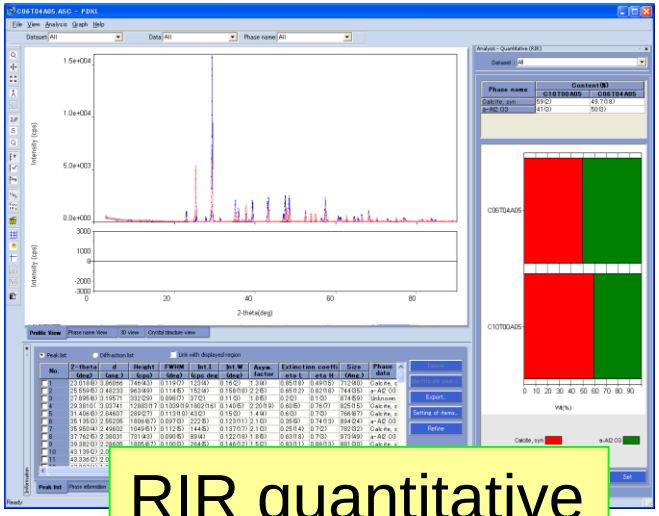


# PDXL Basic Package

- **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.



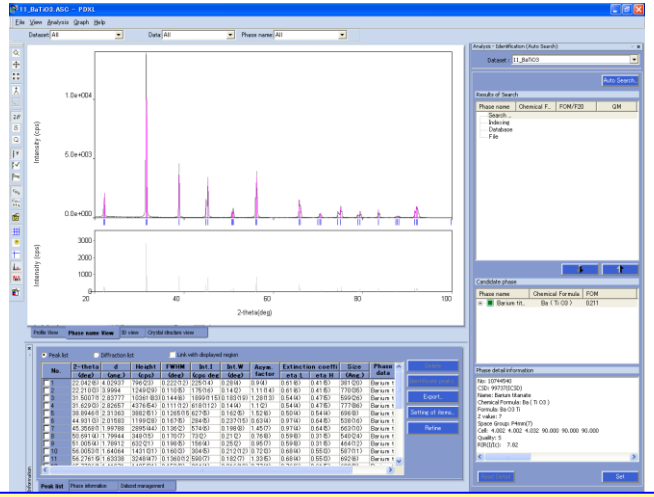
Peak edit



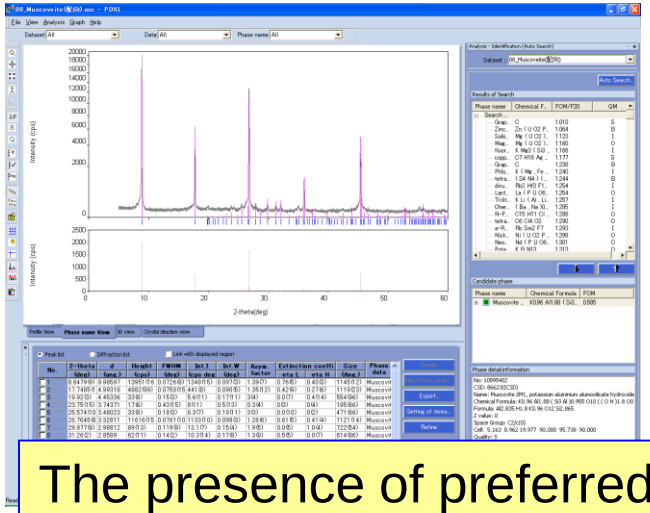
RIR quantitative

# 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):

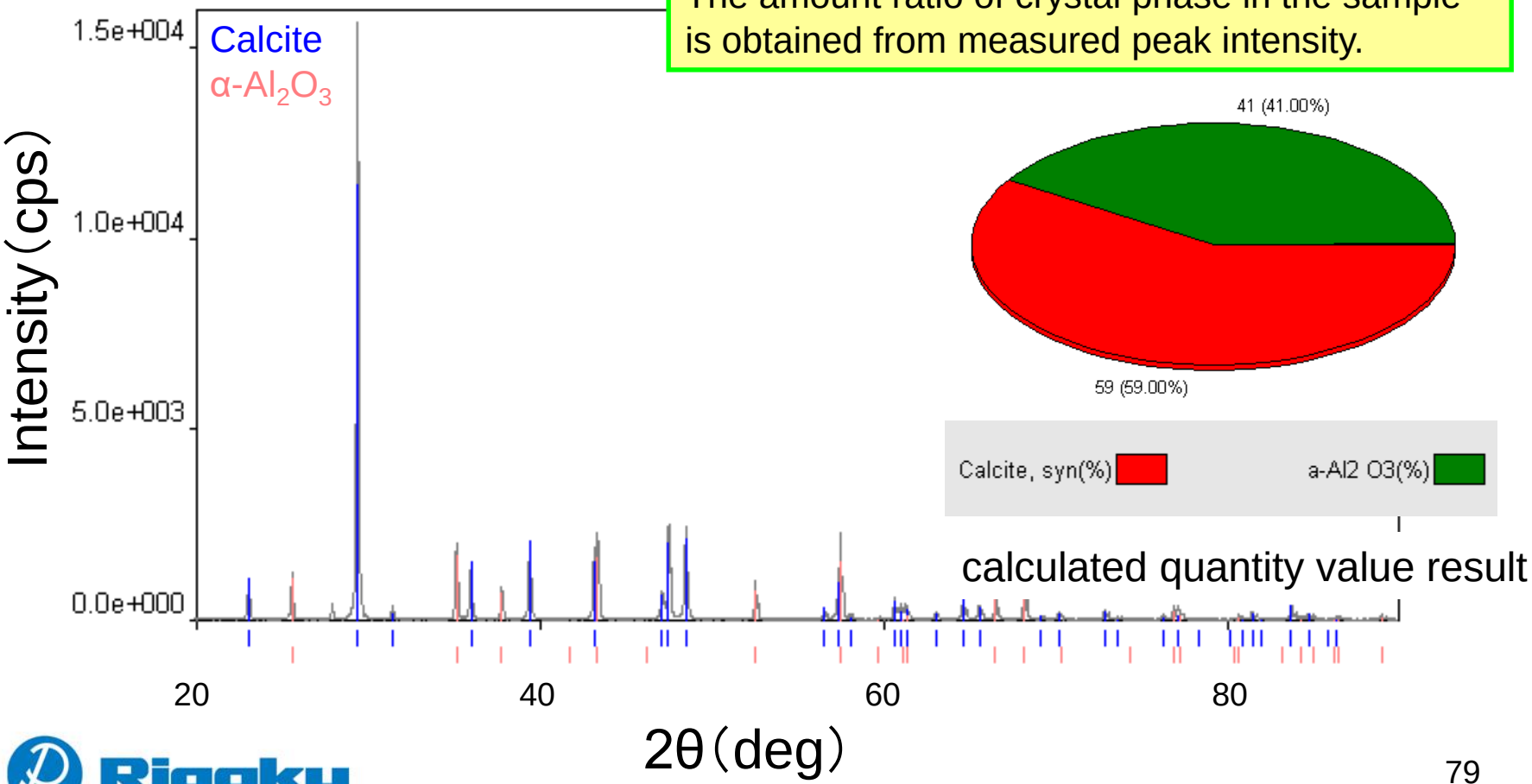
C:\ProgramData\Rigaku\Database\PDXL2\USER

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

# Quantitative Analysis

- How much of each phase is there?

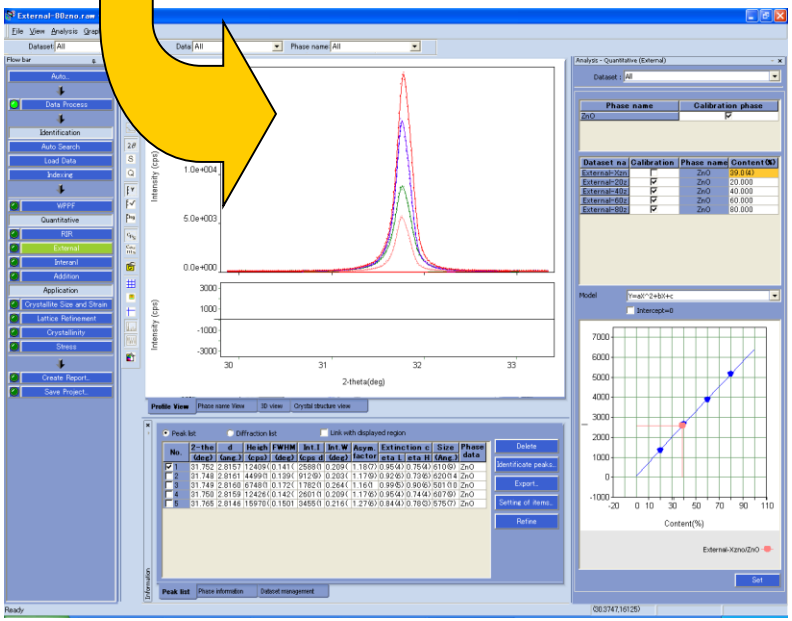
The amount ratio of crystal phase in the sample is obtained from measured peak intensity.

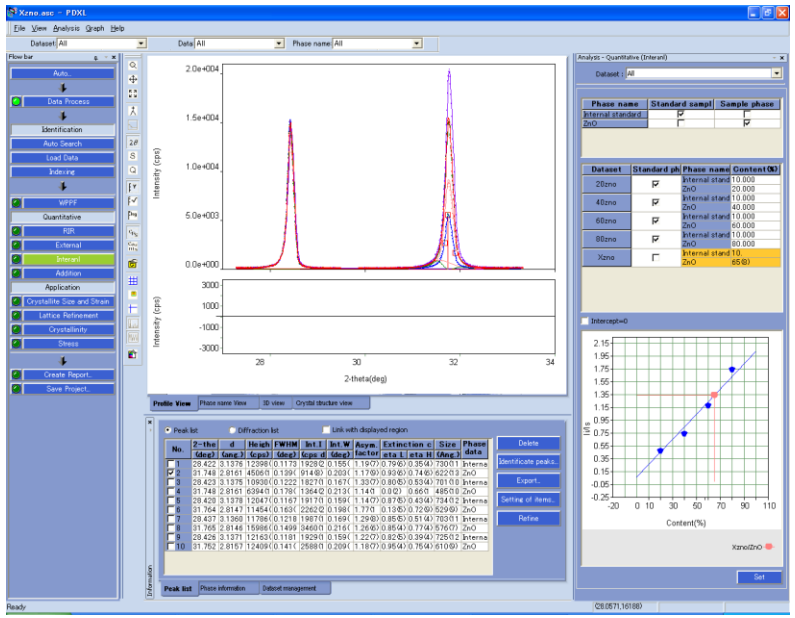


# PDXL: Quantitative Analysis

- 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.

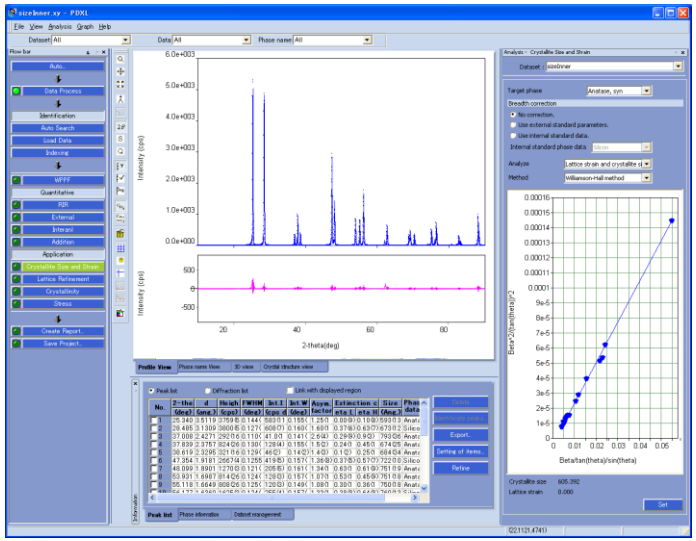
It is convenient for the process control etc.

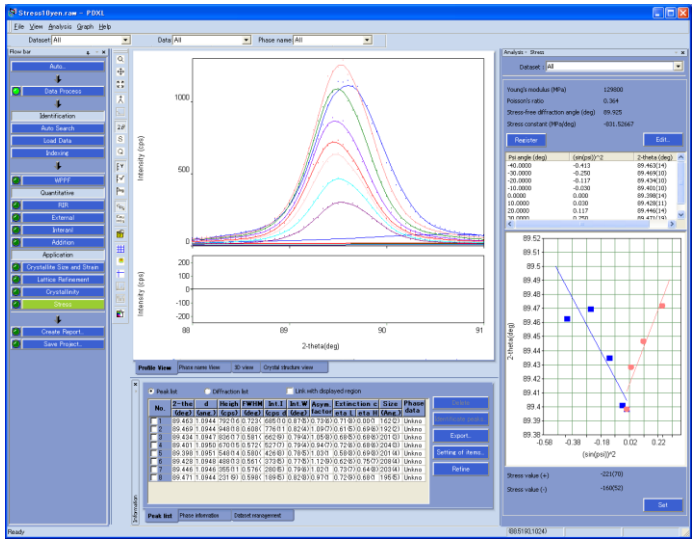




# PDXL: Application Analysis

- 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 by estimated in very easy way by using Scherrer equation (included in PDXL basic):

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

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).