Introduction to Winplotr and FullProf

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Outline

- General overview of FullProf suite of programs
- Winplotr program
- Introduction to PCR-files and PCR-editor
  - Indexing & extracting
  - Mix of known and an unknown phases
  - Fourier analysis to complete the structure

General overview of FullProf suite of programs

- The FullProf Suite is formed by a set of crystallographic programs mainly developed for Rietveld analysis of neutron or X-ray powder diffraction data collected at constant or variable step in scattering angle.
- Main author: J. Rodríguez-Carvajal (ILL)
- Website: http://www.ill.eu/sites/fullprof/
- MS Windows, Mac OS, Linux.

General overview of FullProf suite

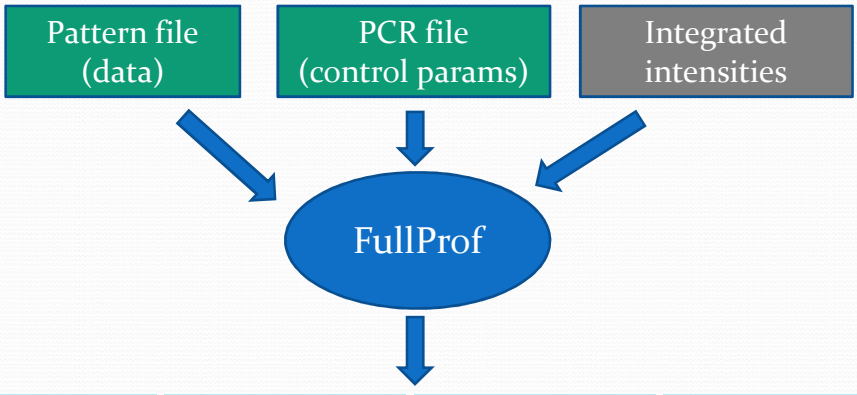
- FullProf
  - Structure refinement (Rietveld, Montecarlo, Simulated annealing)
  - Diffraction pattern simulation (calculation)
  - Profile matching (intensity extraction)
  - Powder patterns and single crystals
  - Multiple phases
  - Multiple patterns
  - Incommensurated structures
  - Magnetic diffraction (powder, SX, polarized neutrons)

Can mix different jobs and different data sets

General overview of FullProf suite

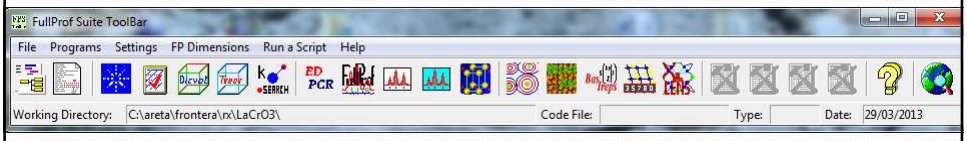
- FullProf (Rietveld)
  - Multiple phases and/or patterns.
  - Powerful microstructure analysis (anisotropic effects)
  - Different functions to describe peak profiles and asymmetries
  - Geometry calculations (and BVS analysis)
  - Different ways to describe the structure:
    - Atomic positions
    - Rigid bodies
    - "Amplimodes" (amplitude of vibrating modes)
  - Sequential running on several input files

General overview of FullProf suite



General overview of FullProf suite

- FullProf toolbar
  - Allows launching most of FullProf Suite programs.
  - Working directory
  - Select input files.



- FullProf home page

Winplotr

- Winplotr is a program intended to plot, manipulate and analyze powder diffraction patterns (raw data, normalized data, neutrons, x-rays from lab. or synchr.). Patterns from refinement programs (Rietveld, profile matching, single crystal or extracted intensities refinement), and also other files as microstructural analysis files.
- It is also intended to be a platform for launching other programs of FullProf_suite and other external programs distributed together.

Winplotr

- Winplotr (old, new)
  - Plotting data: CW or TOF
  - Multiple patterns
  - Different choices of x-axis (2θ, Q, 1/d, d,…)
  - Plenty of tools (zoom, cursor position, scrolling)
  - Tools for analysis : background selection, peak search, profile fitting, peak integration, smoothing …
  - Plotting Rietveld refined data (I-obs, I-cal, difference, Bragg positions)
  - Launching other applications (FullProf, indexing programs, GFourier, FpStudio…)

General overview of FullProf suite

- Fourier & Gfourier
  - Fourier program makes Fourier analysis: Fourier transform structure factors using calculated phases to obtain real-space electron density.
  - GFourier interface to custom Fourier calculations and a tool for displaying the Fourier analysis.
    - Patterson
    - Observed, calculated, observed – calculated density
    - Peak search / identification
    - Geometric calculation (peak distances)

General overview of FullProf suite

- FullProf Studio
  - Drawing the structure of the compounds
  - Bonds and polyhedra
  - "Amplimodes"
  - Magnetic structures

Introduction to PCR files

- Input parameters for FullProf.
- Complexity inherent to the large number of parameters controlling the refinement:
  - Measuring conditions (geometry, wavelength(s), radiation, …)
  - Procedure desired (Rietveld, profile matching,…)
  - Data format, excluded regions, types of corrections
  - Zero error, other corrections , background.
  - Phases information: space group, atomic positions, lattice parameters.
  - Profile description and FWHM parameters…

PCR file

```
La0.625Ca0.375MnO3 RX   P nma  RT
NPATT      1
W_PAT   1.000
!Nph Dum Ias Nre Cry Opt Aut
   1   1   1   0   0   0   1
!Job Npr Nba Nex Nsc Nor Iwg Ilo Res Ste Uni Cor
   0   7   0   2   0   0   0   0   0   0   0
!File names of data(patterns) files
lcm625.gss
!
!Mat Pcr Syo Rpa Sym Sho
   1   1   0   0   1   0
!Ipr Ppl Ioc Ls1 Ls2 Ls3 Prf Ins Nkl Fou Ana
   0   0   1   0   0   0   3  12   0   2   1
!
! lambda1 Lambda2    Ratio     Bkpos    Wdt    Cthm     muR   AsyLim   Rpolarz ->Patt# 1
 1.540560 1.544390 -0.4500   40.000 25.5000  0.5800  0.0000   34.00    0.0000
!
!NCY  Eps  R_at  R_an  R_pr  R_gl
  4  0.30  1.00  1.00  1.00  1.00
!     Thmin        Step        Thmax     PSD     Sent0  -> Patt#: 1
    19.0000     0.0300   120.0100   0.000   0.000
```

no. of patterns

no. of phases

X-rays, neutrons, simulation

Background description

Data file format

Wavelengths

Convergence criterion; damping factors.

Introduction to PCR files

```
!
! Excluded regions (LowT  HighT) for Pattern#  1
    -20.30       10.00
    120.00      180.00
!
!
     26    !Number of refined parameters
!
!  Zero     Code    Sycos    Code   Sysin    Code  Lambda     Code MORE ->Patt# 1
-0.03291  21.00  0.00000    0.00  0.00000    0.00 0.000000    0.00    0
!   Background coefficients/codes  for Pattern#  1
 44.848     -51.479      61.393     -37.813      9.0953      0.0000
      0.000       0.000       0.000       0.000       0.000       0.000
!-------------------------------------------------------------------------------
!  Data for PHASE number:   1  ==> Current R_Bragg for Pattern#  1:     3.54
!-------------------------------------------------------------------------------
```

Excluded regions

Zero error

Background (polynomial)

Introduction to PCR files

```
La0.625Ca0.375MnO3
!
!Nat Dis Ang Jbt Isy Str Furth      ATZ    Nvk More
   5   0   0   0   0   0   0   819.1300    0   1
!Jvi Jdi Hel Sol Mom Ter
   1   3   0   0   0   0
!Contributions (0/1) of this phase to the  1 patterns
 1
!Irf Npr Jtyp  for Pattern#  1
   0   7   0
! Pr1    Pr2    Pr3    Brind.   Rmua   Rmub   Rmuc     for Pattern#  1
  0.000  0.000  1.000  1.000  0.000  0.000  0.000
!
! Max_dst(dist) (angles)  Bond-Valence Calc.
      3.0000      2.0000        AAA
P n m a                  <--Space group symbol
!Atom Typ       X         Y         Z      Biso      Occ      In Fin N_t Spc /Codes
 La   La     0.01843   0.25000   0.00318  0.69705  0.31250   0   0   0    0
               51.00      0.00     61.00    81.00     0.00
 Ca   CA     0.01843   0.25000   0.00318  0.69705  0.18750   0   0   0    0
               51.00      0.00     61.00    81.00     0.00
```

Number of atoms in the asymmetric unit

Function describing the profile

Atom labels

Symbol

Atomic positions

Debye-Waller, occupancy

Introduction to PCR files

Scale factor

FWHM parameters

```
!-------> Profile Parameters for Pattern #  1
!  Scale          Shape1       Bov        Str1       Str2       Str3    Strain-Model
 0.53863E-03   0.00000   0.00000   0.00000   0.00000   0.00000        0
    0.00000     0.000     0.000     0.000     0.000     0.000
!      U          V          W          X          Y        GauSiz   LorSiz Size-Model
  0.004032  -0.013992   0.007475   0.159584   0.000000   0.001151  0.000000     0
   171.000    181.000    161.000     11.000      0.000     21.000     0.000
!     a          b          c        alpha       beta       gamma
  5.447907   7.693172   5.462159  90.000000  90.000000  90.000000
  51.00000   41.00000   31.00000    0.00000    0.00000    0.00000
!   Pref1      Pref2      Asy1       Asy2       Asy3       Asy4        S_L        D_L
  0.00000   0.00000   0.02728   0.01962   0.00000   0.00000   0.00000   0.00000
     0.00      0.00      0.00      0.00      0.00      0.00      0.00      0.00
!Additional U,V,W parameters for Lambda2
  0.008390  -0.015314   0.008201   <--  U2,V2,W2 for lambda(2)
  0.000000   0.000000   0.000000
```

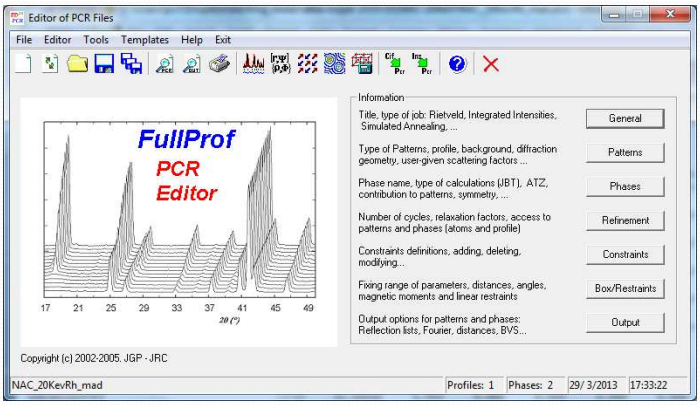
Cell parameters

Preferred orientation parameters

Assymetry parameters

Introduction to PCR files

- Start with a similar PCR (included)
- Modify it for the desired job



FullProf CodeWords

```
P n m a                    <--Space group symbol
!Atom Typ      X         Y         Z        Biso      Occ      In Fin N_t Spc /Codes
Mn    MN     0.00000   0.00000   0.50000   0.50550   0.50000   0   0   0   0
                0.00      0.00      0.00     91.00      0.00
La    La     0.01824   0.25000   0.00292   0.67937   0.31250   0   0   0   0
               61.00      0.00     71.00     81.00    101.00
Ca    CA     0.01824   0.25000   0.00292   0.67937   0.18750   0   0   0   0
               61.00      0.00     71.00     81.00   -101.00
```

Control parameters to be refined and the constrains between them.

Fixed format (i.e. non scientific).

|Code|/10 → integer part: Parameter no.

Residual × sign: Damping factor

FullProf CodeWords

```
P 63 m c                    <--Space group symbol
!Atom   Typ        X         Y         Z      Biso       Occ      In Fin N_t Spc /Codes
!    beta11    beta22    beta33    beta12    beta13    beta23   /Codes
P       P       0.33333   0.66667   0.75851   0.00000   0.16667    0   0   2    0
                  0.00      0.00     51.00      0.00      0.00
        0.03448   0.03448   0.02232   0.01724   0.00000   0.00000
         111.00    111.00    131.00    110.50      0.00      0.00
O1      O       0.37332   0.18668   0.31044   2.15000   0.50000    0   0   0    0
                  41.00     40.50     61.00      0.00      0.00
O2      O       0.33333   0.66667   0.57077   2.15000   0.16667    0   0   0    0
                  0.00      0.00     71.00      0.00      0.00
```

Oxygen O1 is at 6*c* Wyckoff position of *P* 63 *m c* that can be expressed as:
(2*x*, *x*, *z*)
41.00 40.50

Occupancy factors

```
P n m a                     <--Space group symbol
!Atom Typ        X         Y         Z      Biso       Occ      In Fin N_t Spc /Codes
Mn    MN      0.00000   0.00000   0.50000   0.50550   0.50000    0   0   0    0
                  0.00      0.00      0.00     91.00      0.00
La    La      0.01824   0.25000   0.00292   0.67937   0.31250    0   0   0    0
                 61.00      0.00     71.00     81.00      0.00
Ca    CA      0.01824   0.25000   0.00292   0.67937   0.18750    0   0   0    0
                 61.00      0.00     71.00     81.00      0.00
```

Due to the procedure that FullProf uses for the calculus of structure factor, atomic occupancies must be scaled with the multiplicity of the Wyckoff position of the atom: $La_{0.625}Ca_{0.375}MnO_3$:
P nma: General position (8*d*: multiplicity 8)
Mn: (4*b*) 0 0 ½ → occupation factor 1×4/8= 0.5
La: (4*c*) *x* ¼ *z* → occupation factor 0.625×4/8= 0.3125
Ca: (4*c*) *x* ¼ *z* → occupation factor 0.375×4/8= 0.1875

Indexing (Winplotr & Dicvol, Treor, Ito)

- Automatic peak search
  - Check the result manually (adding and/or removing peaks)
- Save points as input files for indexing programs (dicvol, treor or ito)
- Running the selected indexing program
- Automatic generation of a pcr file for profile matching

Patterns with more than one phase

- FullProf can handle multiple phases and with different type of refinements:
  - Rietveld
  - Profile matching (intensity extraction)
  - Profile matching (with fixed intensities)
- Usefull when mixing known and an unknown phases.
  - Example: mineral sample measured in transmission mode through a substrate.
  - In some regions two phases: Aerinite + an unknown phase (Illerdite)

Completing the structure by Fourier analysis

- Refine data with the partial structure (FullProf)
- Prepare files to desired Fourier synthesis (Obs, or Obs-Calc density)
- Search for peaks away from atomic positions.
  - Example: pcr file corresponding to a perovskite with a missing oxygen.

Documentation

(When all else fails, read the instructions)

AN INTRODUCTION TO THE PROGRAM

FullProf

2000

(Version July2001)

FullProf News
Winplotr
EDPCR Manual
Fp-Studio
Fourier

Thanks for your attention