Online Full Profile Search Match and the Crystallography Open Database

Luca Lutterotti[a,b], Daniel Chateigner[b], Philippe Boullay[b], Henry Pilliere[c], Christophe Fontugne[c]
[a]Dipartimento di Ingegneria Industriale, Università degli Studi di Trento, Italy
[b]CRISMAT-ENSICAEN, CNRS UMR 6508, Université de Caen-Basse Normandie, Caen, France
[c]INEL SAS, Artenay, France

The NANOAIR project - granted by FP7-RTD (Grant agreement N° 222333)

The air quality is one of the major concerns for health. There is a need for continuous monitoring of the particulate especially in the submicron range. XRD is one of the principal techniques for the analysis of powders and fundamental to identify dangerous products. An European project named Nanoair has been concluded successfully which aim was to develop a mobile instrument for qualitative and quantitative analysis of breathable particles. The instrument uses XRD and SAXS for the characterization of the automatically collected samples.

Goal of the project and general idea

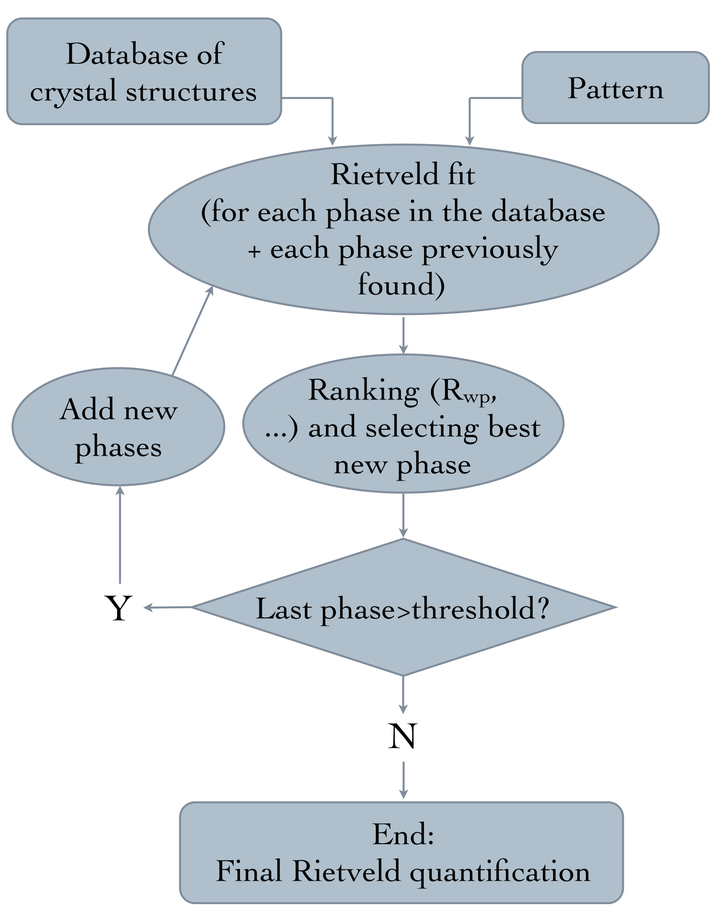
Build a complete portable system that collect the dust, analyses it in automatic to obtain particle sizes (by SAXS), phase identification and quantification along with crystallites/microstrains (by XRD) and finally store all data and results online and the samples locally for reference. For the XRD automatic analysis objective, we recognized:

- Actual search-match softwares require still human intervention to judge which phases are to be accepted or rejected after identification
- The only quantification method easily automatizable and reliable for such task is the Rietveld method but we are limited to the phases with a structure already refined (and deposited in one of the databases) unless also the PONKS method is used

The idea is to use the Rietveld (+ PONKS) also for the search-match step (as we are in any case limited to phases usable in the Rietveld).
A Full Pattern Search-Match method has been built and tested using the COD archive (imported to a local database) plus we can add specific phases missing if necessary. COD: www.crystallography.net
The method has been extended also to neutron and electron diffraction. A TOF neutron version is under testing.

The FPSM (Full Pattern Search-Match method)

Developed inside the Nanoair project



Pro:

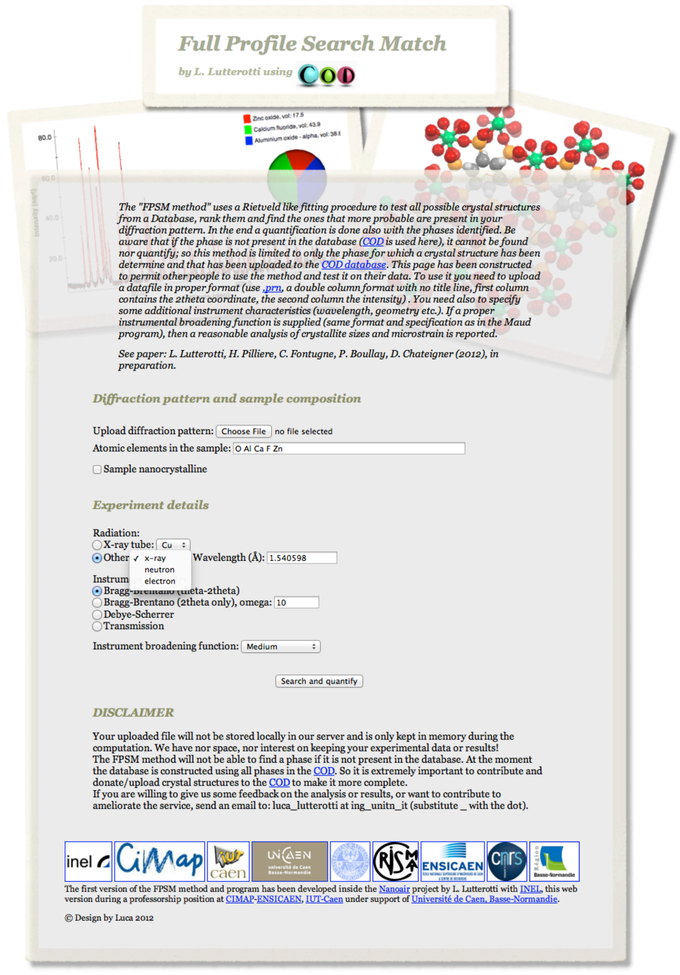
- No user intervention, automatic analysis
- No peaks identification required (works with nano materials/particles
- Full Rietveld quantitative analysis provided
- Works for neutron and electron diffraction

Cons:

- Only phases with know crystal structure are ready to be used (unknown structures require a list of peaks and calibrated intensities, PONKS)
- Available databases are still uncompleted
- If no elemental composition provided → requires > 20 minutes on 12 cores computer
- Good ranking algorithm required for very small phase amount

Further improvements:

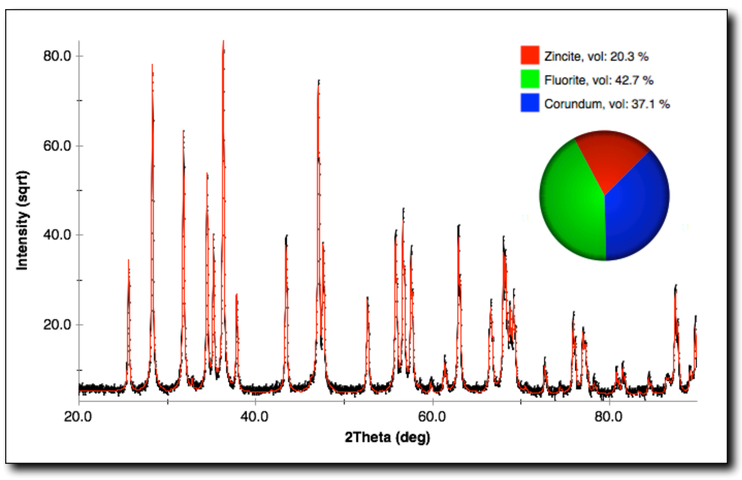
- Combination with fluorescence to remove manual composition restriction



Two trial web pages have been setup online at:
http://nanoair.ing.unitn.it:8080/sfpm
http://cod.iutcaen.unicaen.fr/sfpm
search and quantification is limited by proxy connection timelimit (few minutes) so it should be used restricting the composition as much as possible to speed up computation.

Testing FPSM using the COD

Sample cpd1h of Round-Robin on quantitative analysis
http://www.iucr.org/resources/commissions/powder-diffraction/projects/qarr



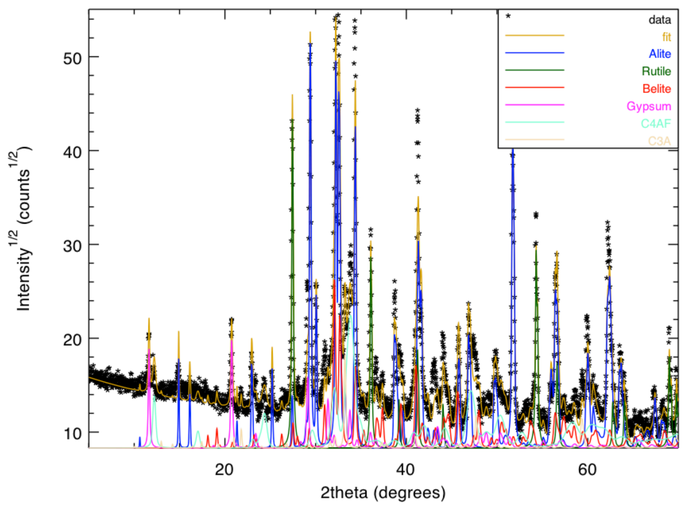
Phase (wt%)	FPSM	Round-Robin
ZnO	29.0	29.6
CaF	33.9	34.3
Al_2O_3	37.1	36.1

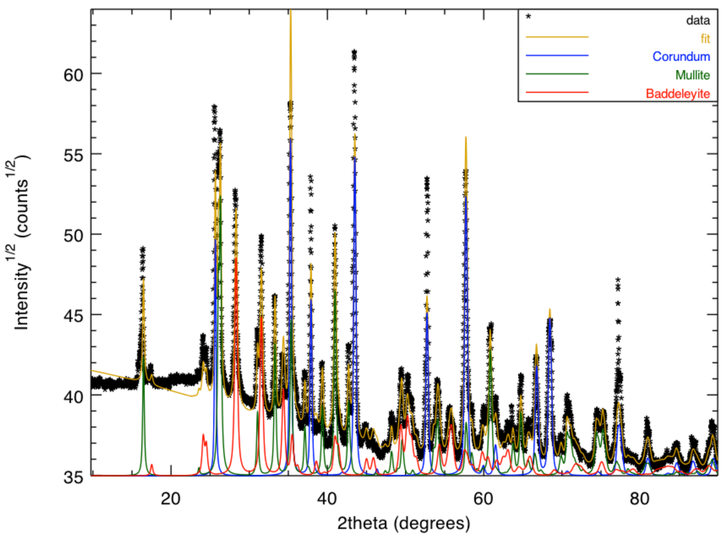
Total computation time (12 cores, 2.93GHz, COD, inorganic):

- No composition restriction: 565 secs
- Only Al, Ca, F, Zn, O, Mg, Na, Si, Cl: 19 secs

Some additional phases (cement phases) has been added to the database as not present in the COD

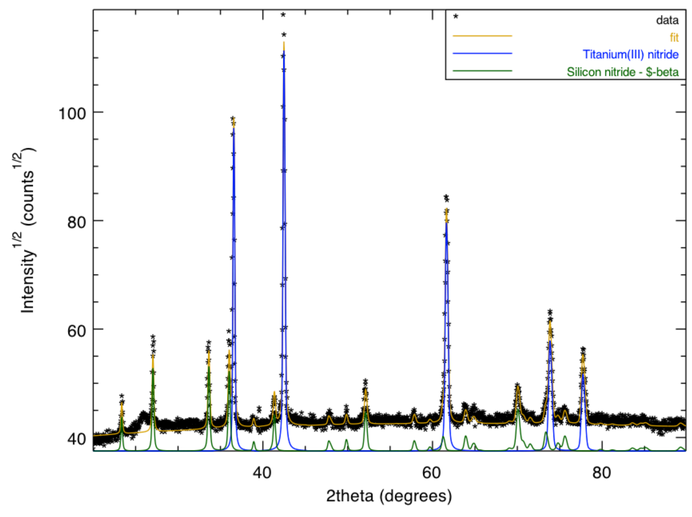
name	vol. (%)	wt. (%)	crystallites (Å)	microstrain
Alite	52.3359	50.1148	2722.86	0.000913623
Rutile	8.7909	11.3199	1382.41	4.9784e-05
Belite	14.6584	14.8578	1014.1	0.00145265
Gypsum	7.28186	5.03503	881.992	0.00175863
C4AF	14.4548	16.3939	485.795	0.00705494
C3A	2.47814	2.27862	1426.2	0.00125916





Phase ID	name	vol. (%)	wt. (%)	crystallites (Å)	microstrain
9009672	Corundum	41.0726	45.1467	730.664	0.00137145
9005502	Mullite	53.5784	46.2318	515.483	0.000527227
9005833	Baddeleyite	5.34905	8.62151	371.805	0.00203445

Final Rietveld analysis, Rw: 0.0455532, GofF: 1.79502



Phase ID	name	vol. (%)	wt. (%)	crystallites (Å)	microstrain
1101045	Titanium(III) nitride	50.0729	62.8891	1682.09	0.00122093
1001246	Silicon nitride - $-beta	49.9271	37.1109	800.99	0.00129495

Final Rietveld analysis, Rw: 0.0601521, GofF: 2.66024