

DE LA RECHERCHE À L'INDUSTRIE



ALGORITHMS FOR ATOM PROBE TOMOGRAPHY CHARACTERISATIONS OPTIMIZATION OF RPV STEELS

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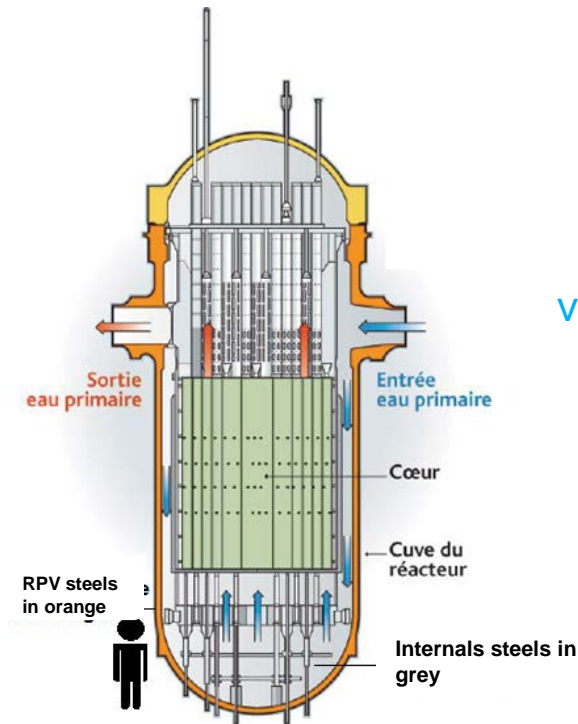
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Nota :This work profited from a French government grant managed by the National Agency of Research under the program "Investments for the future" (ref. ANR-11-EQPX-0020

Anticipate and predict ageing (effects of irradiation) and potential degradation of essential structures (RPV and internals) in PWRs.



Understanding the mechanisms of ageing phenomena at very fine scales (precipitation, solute clustering, segregation to grain boundaries, dislocation loops, black dots, etc.).



TEM

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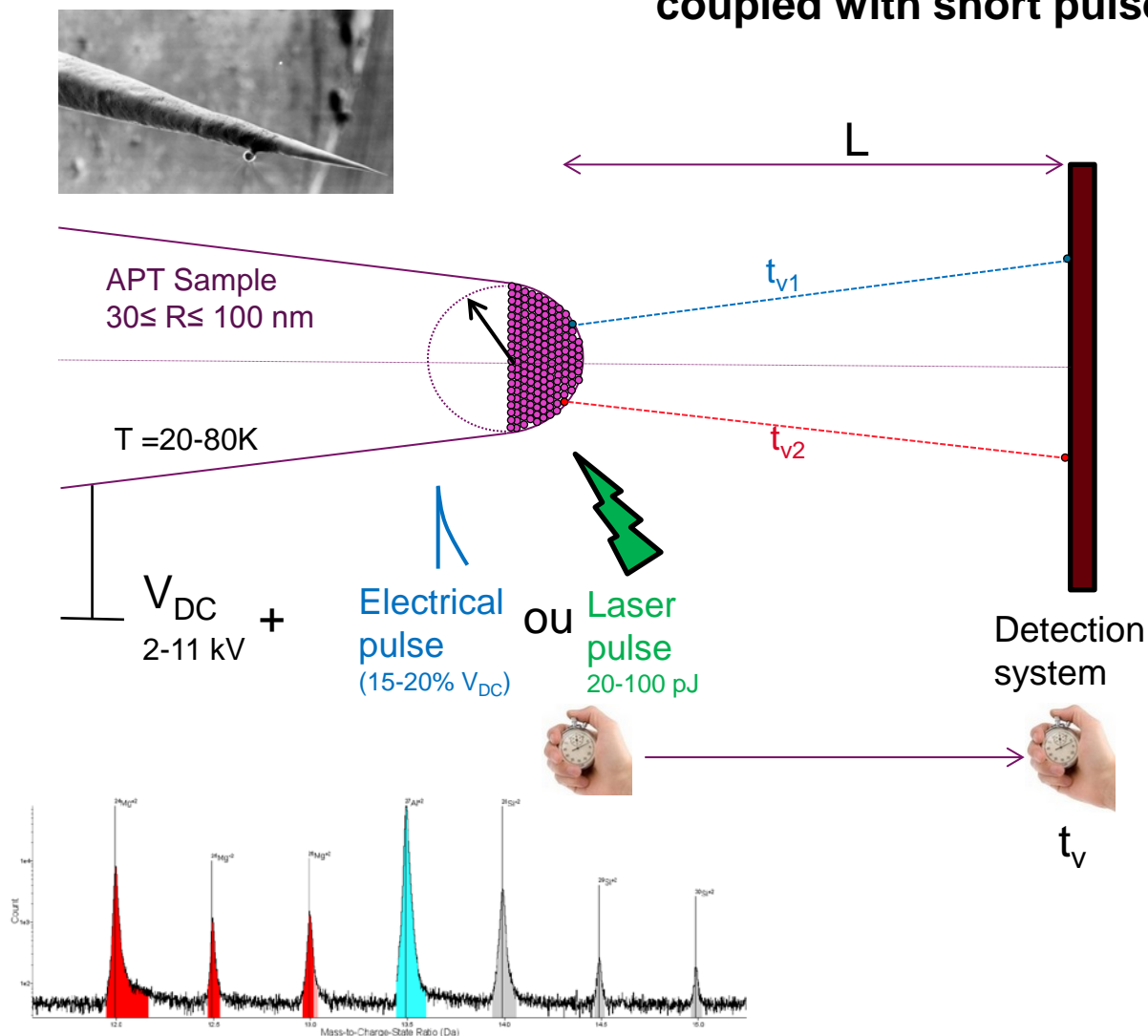


APT (atomic scale)

Crystallographic structure
Size and density of a few tens of nm

Spatial distribution
Chemical composition
Size and density of a few nm
Identification and characterization of nanometric features

Possible evaporation due to the existence of an intense electric field coupled with short pulses



Energie conservation

$$\frac{1}{2} m \left(\frac{L}{t_v} \right)^2 = neV$$



Masse to charge state ratio

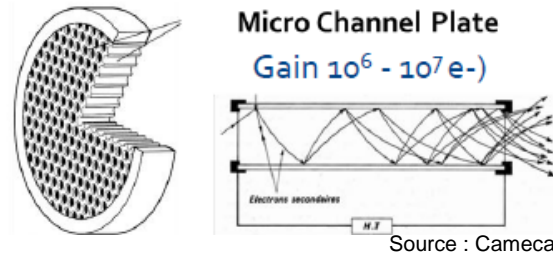
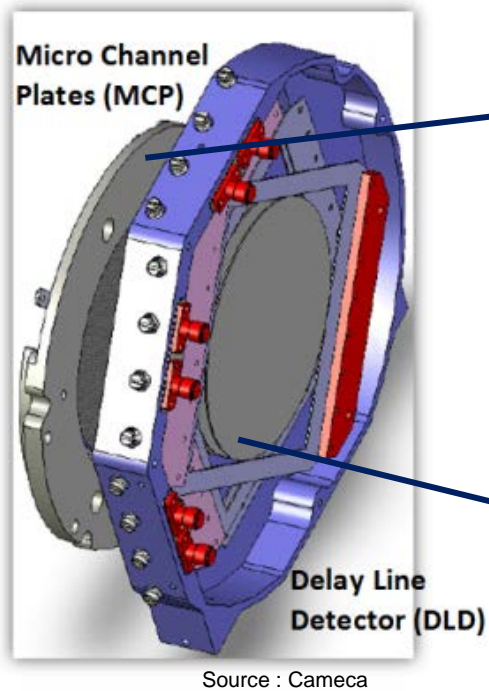
$$\frac{m}{n} = 2eV \left(\frac{t_v}{L} \right)^2$$



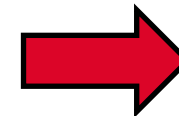
Mass spectrum →

1st information :
Chemical nature of ions

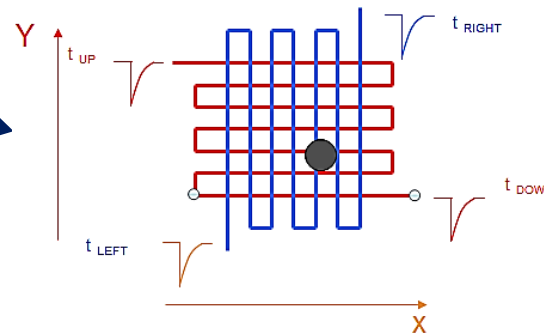
Ion impact in the detection system → Coordinates (X,Y)



MCP : signal amplification composed of a micro channel plate



Ion coordinates in the detector system : (X,Y) + evaporation event

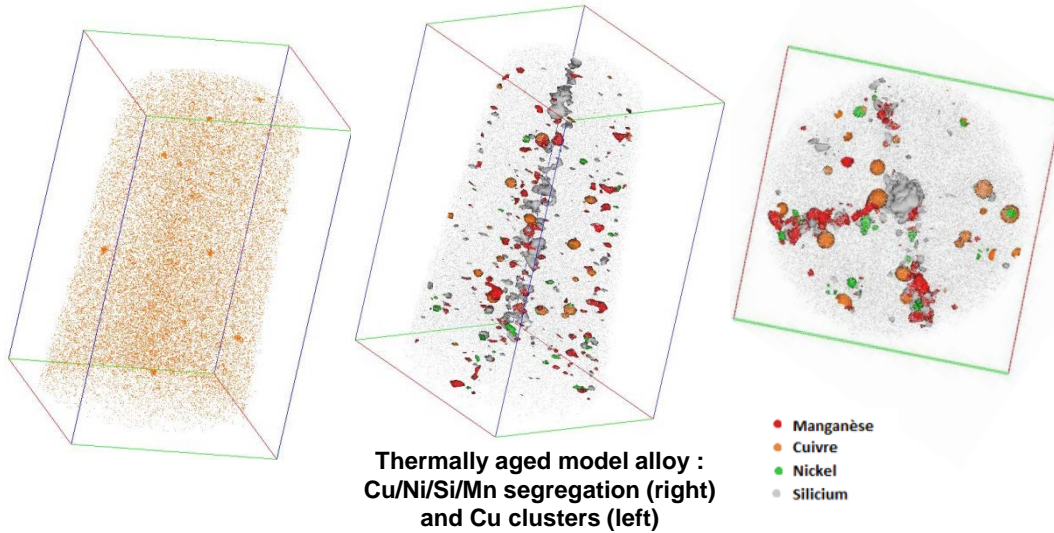


DLD : Delay line detector (grid made from two windings of copper lines)



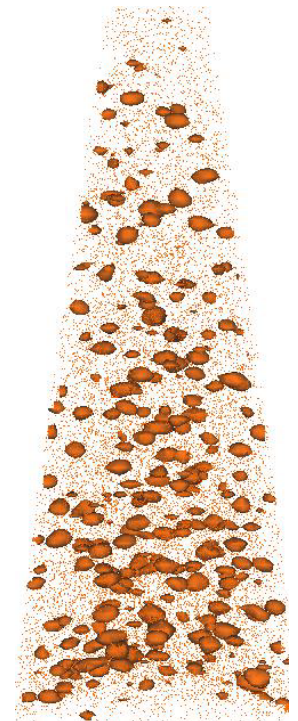
Reconstruction

2nd information :
Atom position
(x,y,z)



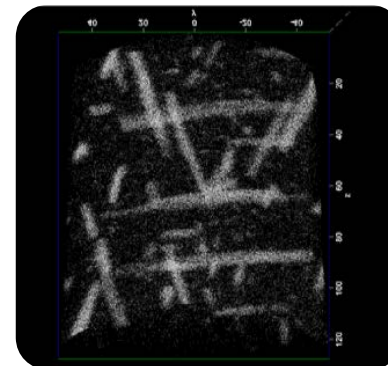
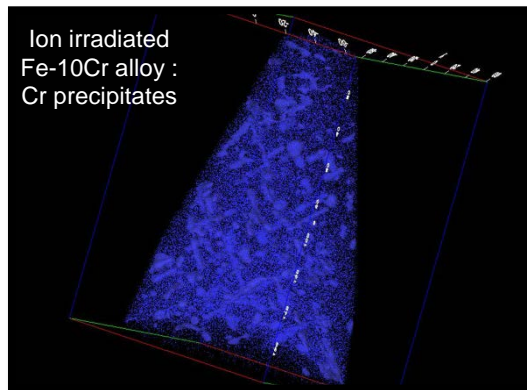
Spatial resolution : 0,4 nm laterally
<0,2 nm en depth
Sensibility : < 5 ppm

CLUSTERING



Proton irradiated
internal steels :
Cu clusters

SEGREGATION



Aluminum alloy 6061-T6 :
MgSi precipitates

PRECIPITATION

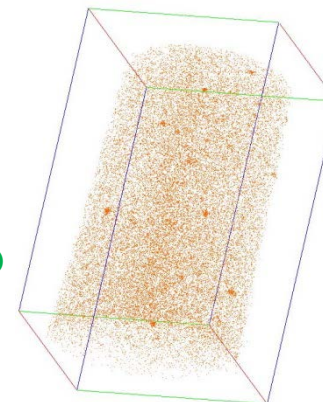
One of the most common method applied to APT data to identify and analyse solute clusters : **Maximum Separation Method (MSM)**

MSM : Relies on 3 parameters **O**, **dmax** and **Nmin**

1st step

2nd step

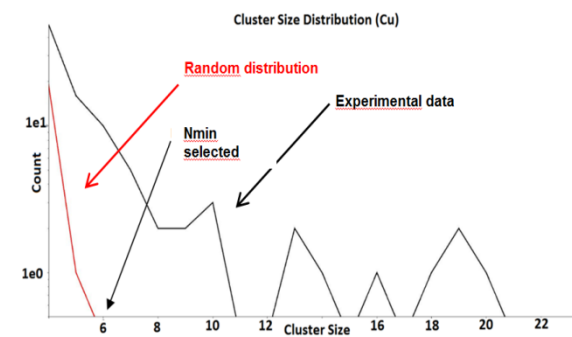
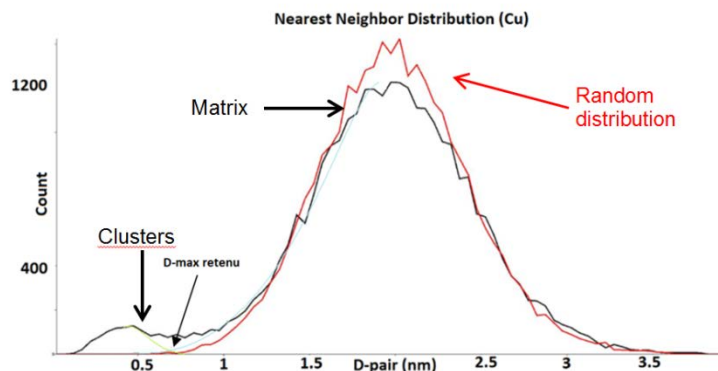
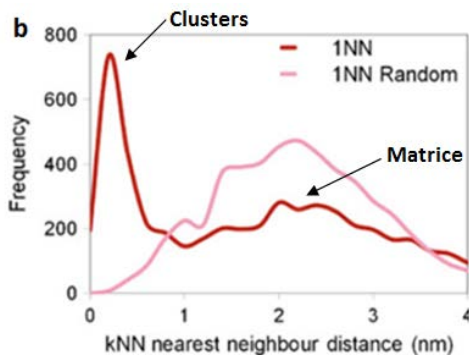
3rd step



Order **O** : number of nearest neighbor considered

dmax : if **O** solute atoms are within the critical distance **dmax** of another solute atom
→ same clusters

Nmin : Minimum number of atoms to avoid random occurrences of a few atoms close together

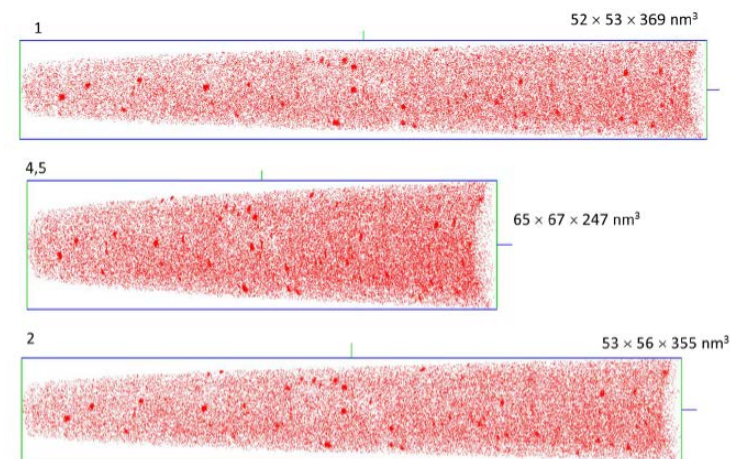
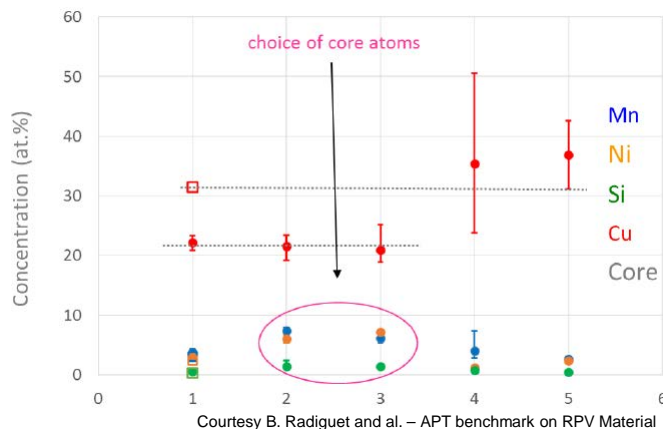


But the results depend strongly on the choice of these parameters !!!

Benchmark on RPV material results: strong influence of identification parameters

	Oder	d_{max} (nm)	N_{min}	L (nm)	$d_{erosion}$ (nm)	Core atoms
1	4	0.9	8	0.7	0.7	Cu
2 (*)	1	0.5	15		-	Cu, Mn, Si, Ni, P
4	4	0.8	6	0.8	0.8	Cu
5	2	0.7	5	0.6	0.6	Cu

Courtesy B. Radiguet and al. – APT benchmark on RPV Material

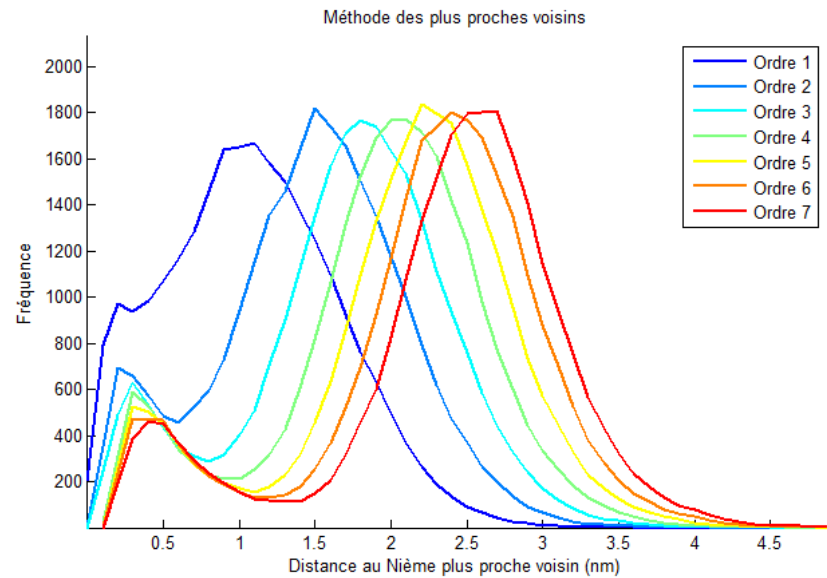


Courtesy B. Radiguet and al. – APT benchmark on RPV Material

To confort and/or help on our identification parameters choice → algorithms

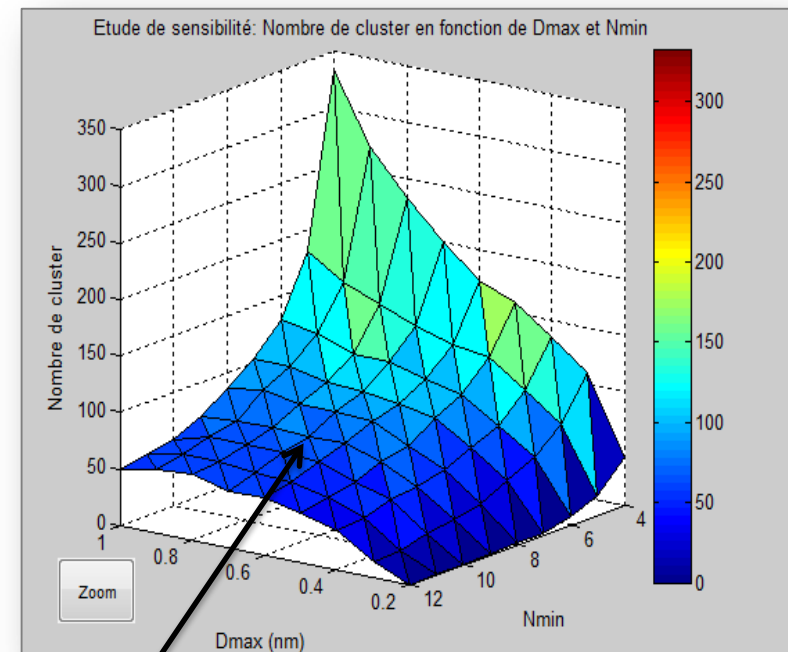
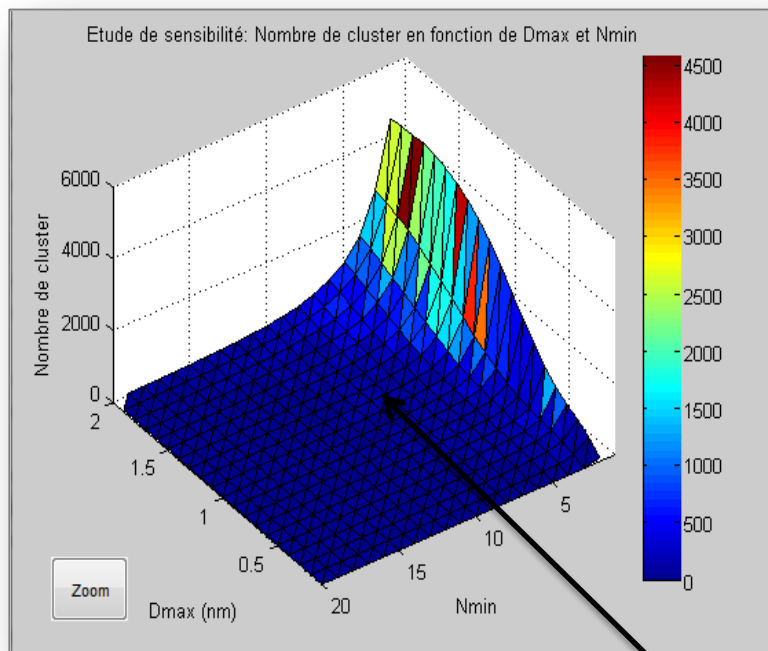
- 1) Optimization of the order
- 2) Sensibility analysis
- 3) Local concentration method

Goal: draw the nearest neighboring solute atoms at different orders in the same g



Allows to choose the most appropriate order more easily and quickly !

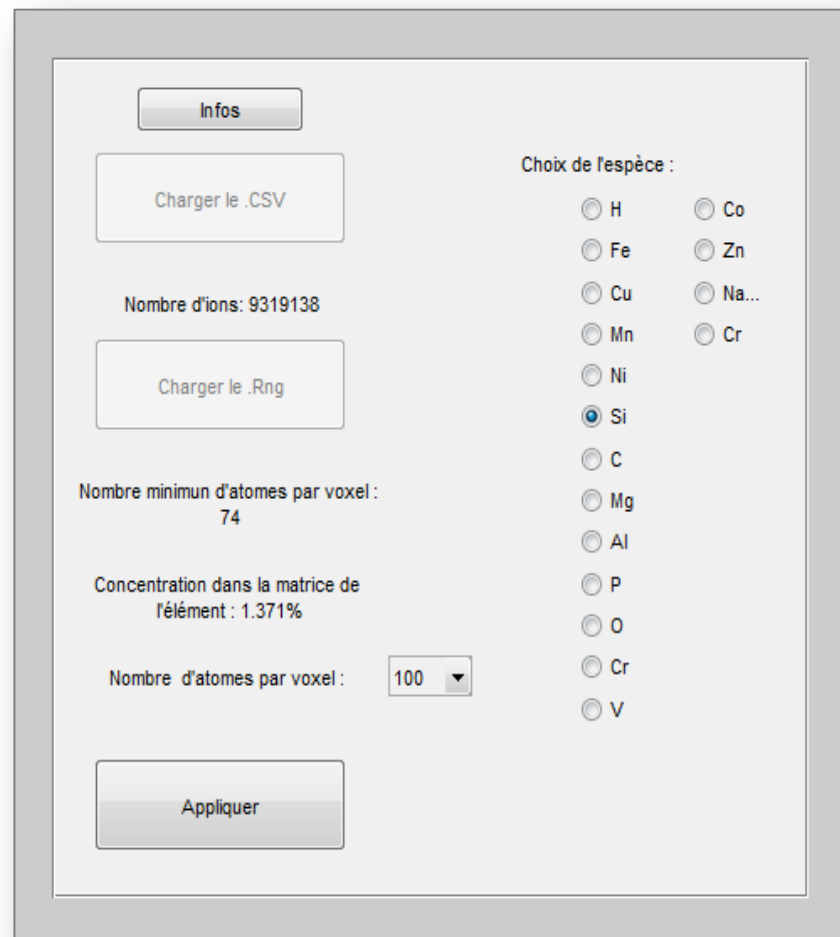
Goal : compare the number of solute clusters found regarding the values of identification parameters d_{\max} and N_{\min} for a fixed order



Wide area of constant cluster density

Sensitivity analysis that should strengthen the choice of identification parameters !

- ❑ Goal : analyze the local solute concentration to increase contrast between features and matrix
- ❑ Method to apply to data with big concentration matrix (high background) before MSM method
- ❑ Useless with weakly concentrated elements



Infos

Charger le .CSV

Nombre d'ions: 9319138

Charger le .Rng

Nombre minimum d'atomes par voxel : 74

Concentration dans la matrice de l'élément : 1.371%

Nombre d'atomes par voxel : 100

Appliquer

Choix de l'espèce :

H Co

Fe Zn

Cu Na...

Mn Cr

Ni

Si

C

Mg

Al

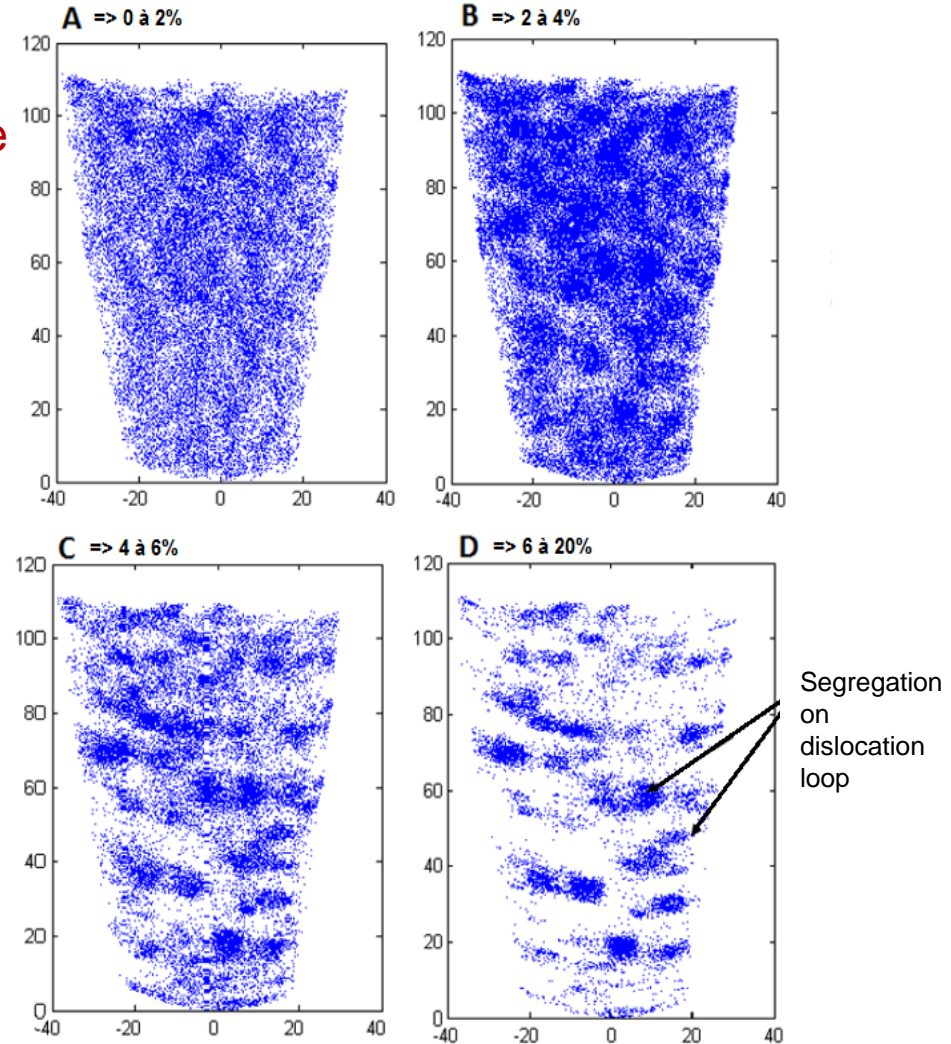
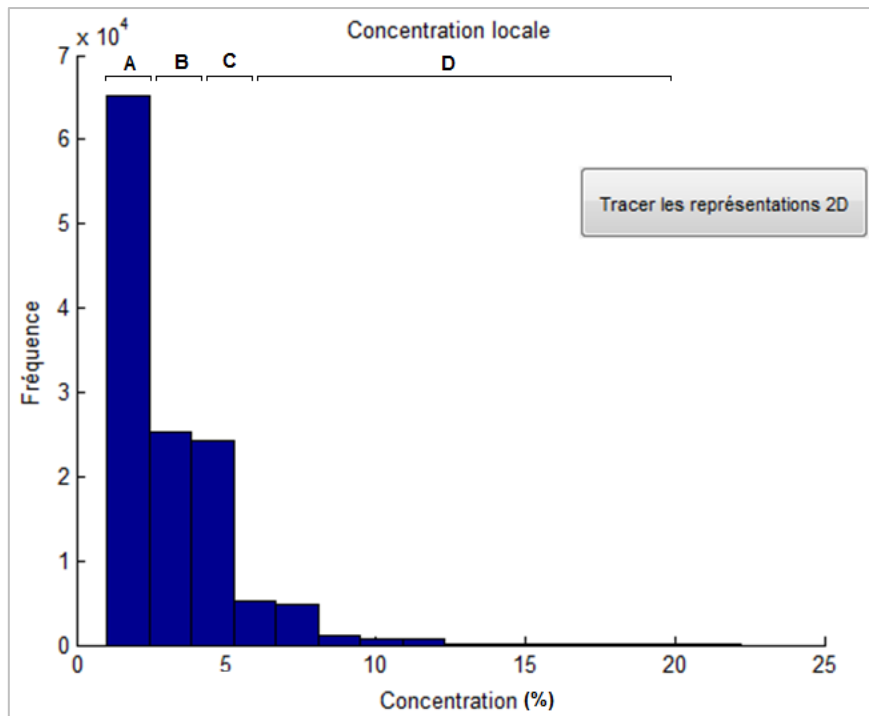
P

O

Cr

V

→ Increase the contrast between features and matrix ! Then the MSM method can be applied in the filtered data to better results



Thanks

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