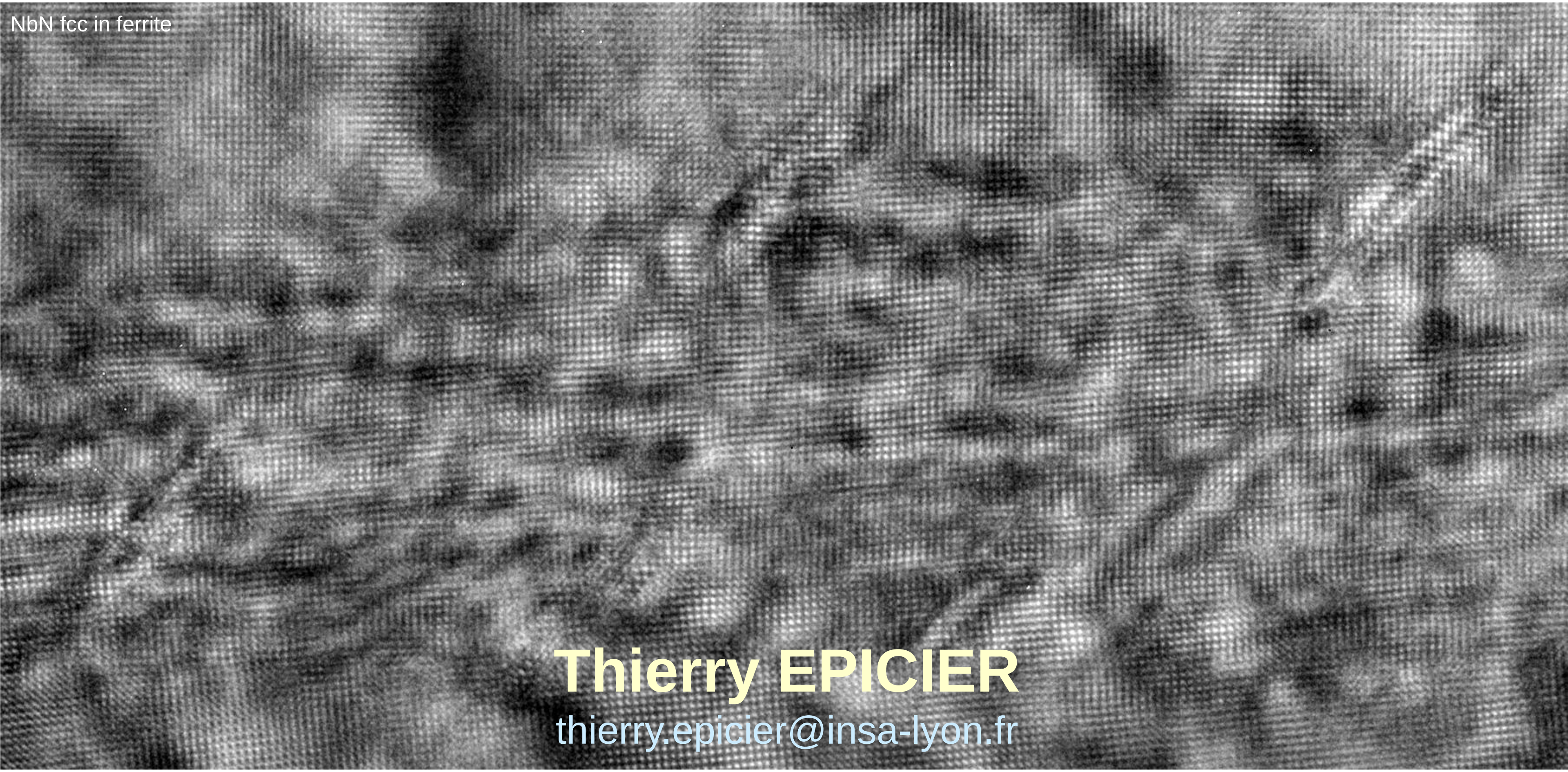
Precipitation of carbo-nitrides in *model* steels



Thierry EPICIER

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Université de Lyon, MATEIS, umr CNRS 5510, INSA-Lyon, Bât. B. Pascal, F-69621 Villeurbanne Cedex

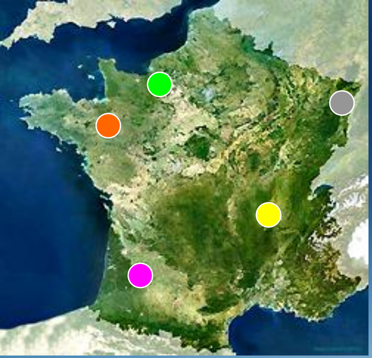
OUTLINE

Brief presentation of INSA - Lyon

Introduction:
context of the work – thermodynamical modelling of the precipitation

Precipitation in the FeNbVC system

Precipitation in the FeNbCN system



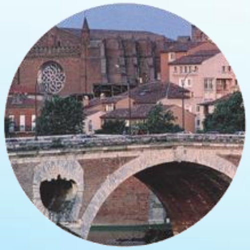
The INSA network

Rennes
Created : 1966
1 600 students

Lyon www.insa-lyon.fr
Created : 1957
5 400 students

Rouen
Created : 1985
1 500 students

Strasbourg
Created : 2003
1 500 students



Toulouse
Created : 1963
2 500 students

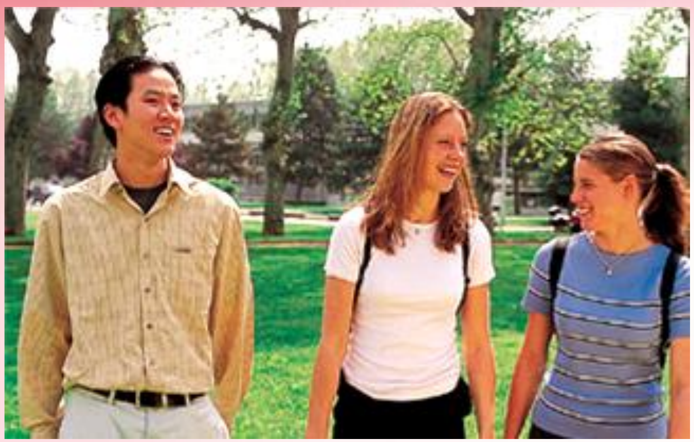
A proactive international politic

- Teaching

  - more than **600 engineering-students abroad** each year

  - welcoming foreign students : **100 nationalities**, **30 % non-french students**

  - **International scheme during 1st cycle** (10 languages taught): **EURINSA, ASINSA, AMERINSA, SCAN**



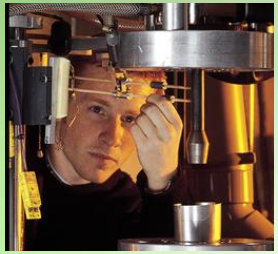
- Research

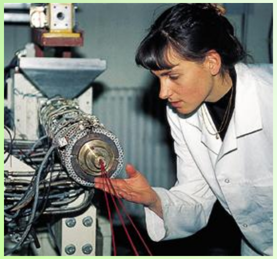
  - over **230 partner universities** around the world

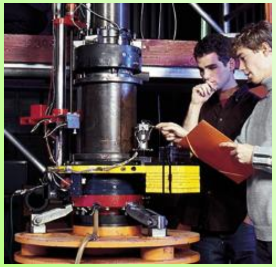
  - **connection Offices** with *Shanghai, Curitiba, São Paulo, Hô-Chi-Minh-Ville, Mexico,* ***Sendaï***

Research at INSA Lyon

A technological research based on Engineering sciences conducted in 20 LABORATORIES

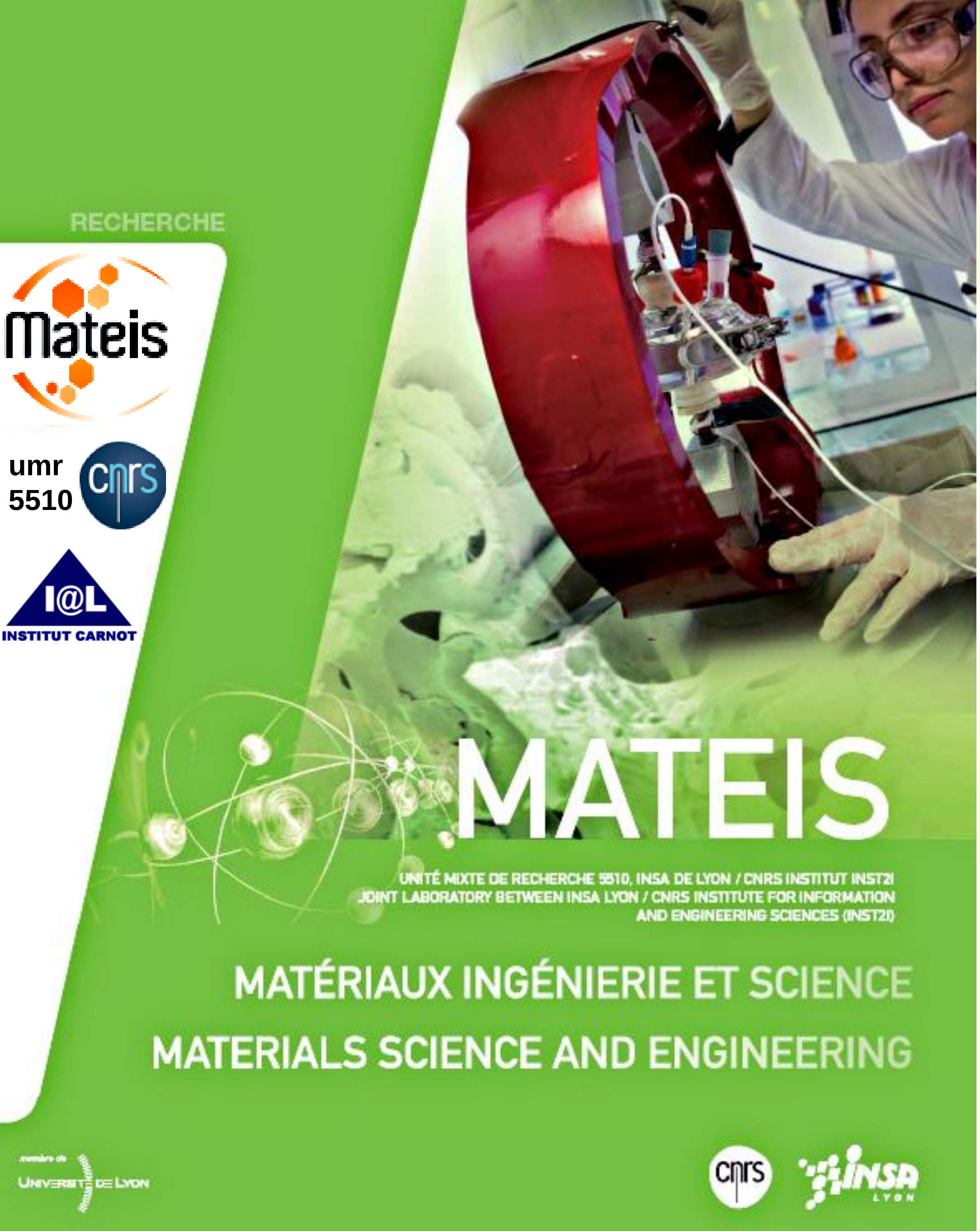






- **Materials Pole :** Functional Materials, Structural Materials, Civil engineering, Metals, Ceramics, Polymers.
- **Mechanics Pole:** Solid Mechanics, Structural Mechanics, Tribology, Acoustics et Vibrations.
- **Energy and Environment Pole :** Security Systems, Waste and Sanitation, Thermal studies, Urban Engineering, Management, Technial Philosophy and Epistemology.
- **Sciences applied to Information and Communication Technologies Pole :** Components and Electronical System, Computer Science, Robotics, Micro and Nano-Technologies, Telecommunications, Treatment of Information.
- **Biology and health Pole :** Health Engineering, Biotechnology, Biochimestry and Pharmacology, Interaction Biology, Biomolecular synthesis, Ethics.

• **Materials Pole:** Functional Materials, Structural Materials, Civil engineering, **Metals, Ceramics, Polymers.**



- **47 permanent academic**
- **7 CNRS researchers**
- **26 technical and administrative**
- **10 Master students**
- **50 to 60 PhD students**
- **14 post-docs**

3 groups by material classes:

Metals and Alloys (METAL)
Prof. J.Y. Buffière

Ceramics and Composites (CERA)
Prof. J. Chevalier

Polymers, Glasses, Heterogeneous Materials (PVMH) Prof. C. Gauthier

3 groups by dedicated know-how:

Structures, Nano-, MicroStructures (SNMS) Prof. T. Epicier*

Interface Reactivity and Corrosion (RI2S) Prof. B. Normand

Durability, Ultrasounds, Instrumented Structures (DUSI) Prof. J. Courbon

*Prof. K. Masenelli since 2010/01

CLYM (Centre Lyonnais de Microscopie)

http://clym.insa-lyon.fr

CETHIL, INSA Lyon

IMP, INSA Lyon / Lyon 1 and St-Etienne Universities

LaMCoS, INSA Lyon

MATEIS, INSA Lyon

CTμ, Lyon 1 University

IRCELYON, Lyon 1 University

LMI, Lyon 1 University

LPCML, Lyon 1 University

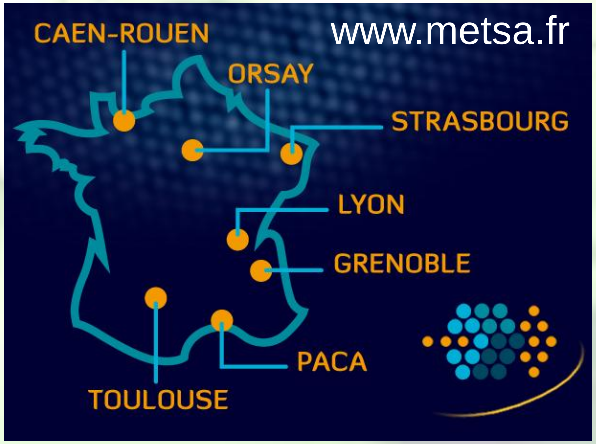
LPMCN, Lyon 1 University

INL, Ecole Centrale Lyon / INSA Lyon / Lyon 1 University

LTDS , Ecole Centrale Lyon

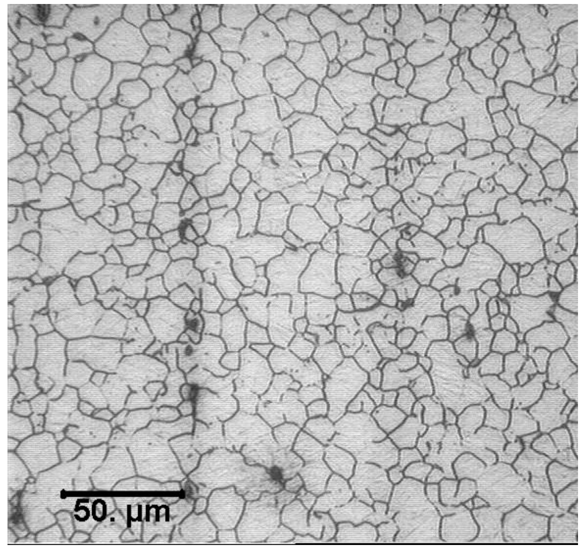
LST, ENS-Lyon

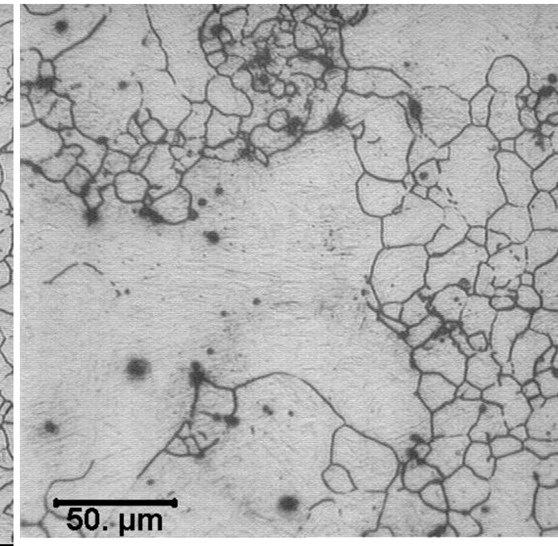
LCC, Ecole Nationale des Mines - St-Etienne



◆ *CONTEXT of the work*

spring steels (Mn/Cr alloyed)
***H**igh **S**trength **L**ow **A**lloyed* **steels** (Nb alloyed)

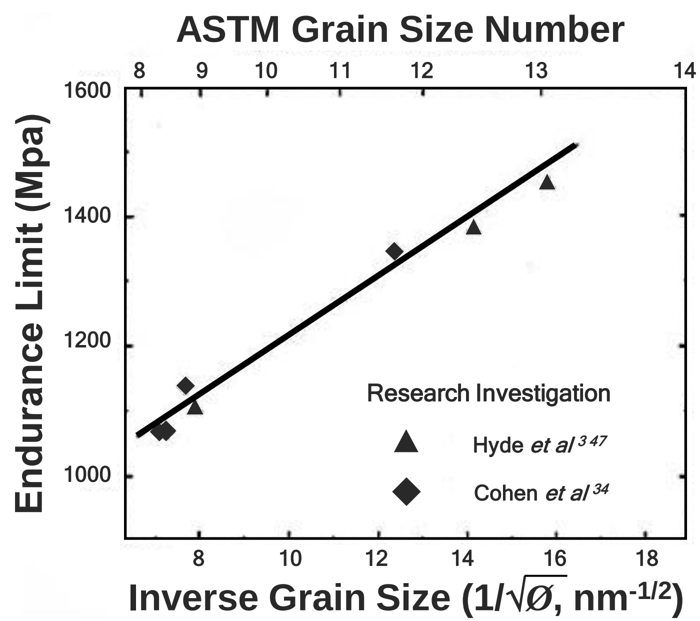




0.02_4 wt. % of NITROGEN **0.01 wt. %**

PRECIPITATION:

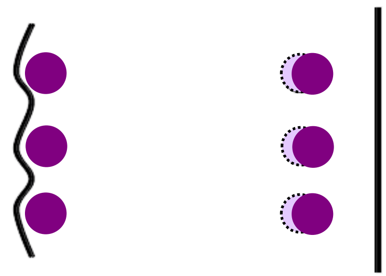
- **Sol. Sol. HARDENING**
- **GRAIN GROWTH control**



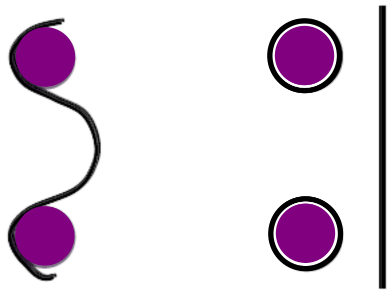
[HYDE et al. *SAE Technical paper,* (1994)]

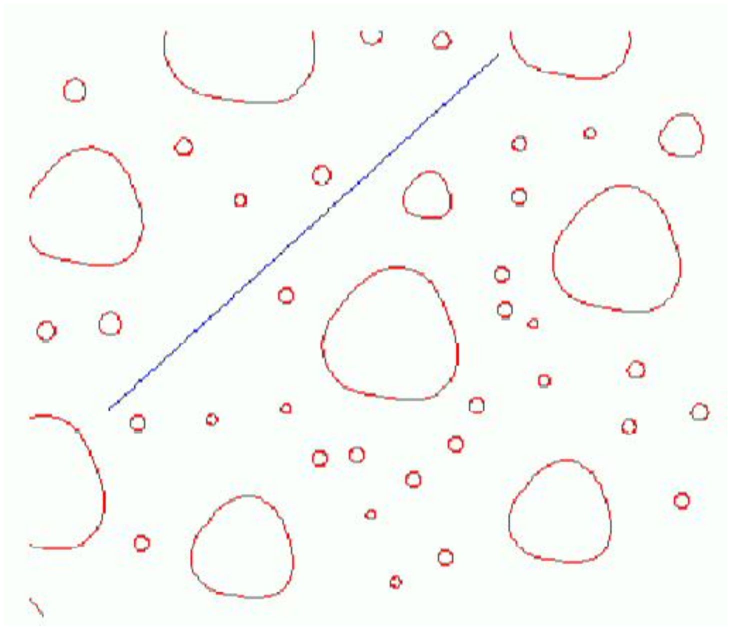
- **Sol. Sol. HARDENING** **(interaction dislocations – precipitates)**

⇨ SHEARING precipitates



⇨ *Orowan mechanism*

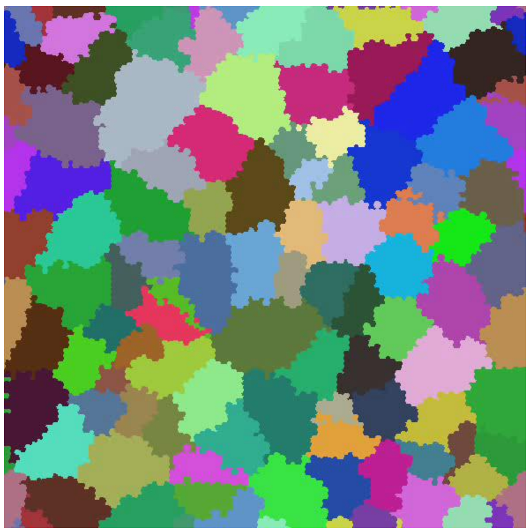




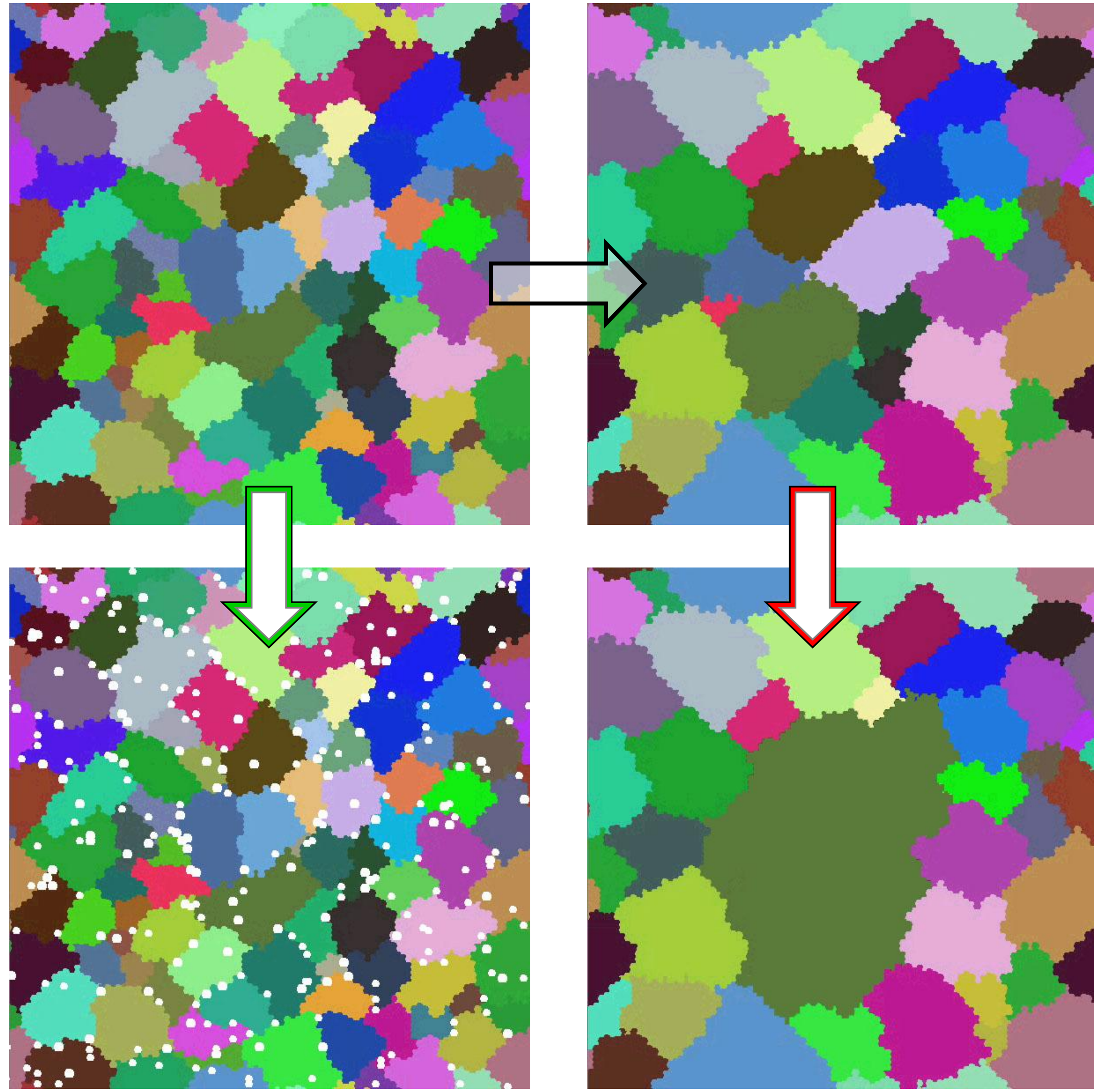
• GRAIN GROWTH control

MECHANICAL RESISTANCE *DECREASES*

when **GRAIN SIZE *INCREASES***



HEAT treatments



PRECIPITATION GRAIN PINNING

ABNORMAL GRAIN GROWTH

THERMODYNAMICAL MODELING of the PRECIPITATION

CLASSICAL NUCLEATION THEORY

1) NUCLEATION *(driving force: **solute supersaturation**)*

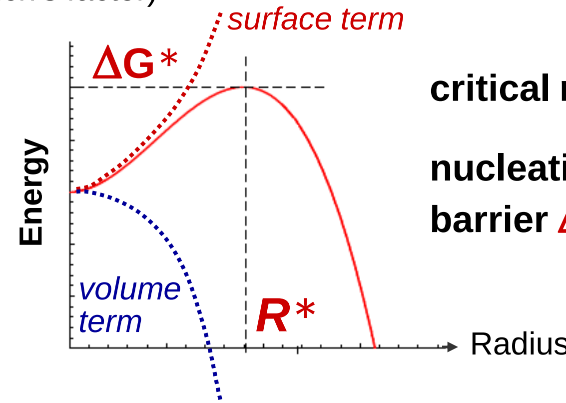
$$\frac{dN}{dt} = N_0 Z \beta^* \exp\left[-\frac{\Delta G^*}{kT}\right]\left(1 - \exp\left[-\frac{t}{\tau}\right]\right)$$

Nucleation rate (N_0: number of nucleation sites per unit volume, τ: incubation time, β^*: condensation rate of monomers, Z: Zeldovich's factor)

$$\Delta G(R) = \underbrace{\frac{4}{3}\pi R^3 \Delta g}_{\text{volume energy}} + \underbrace{4\pi R^2 \gamma}_{\text{surface energy}}$$

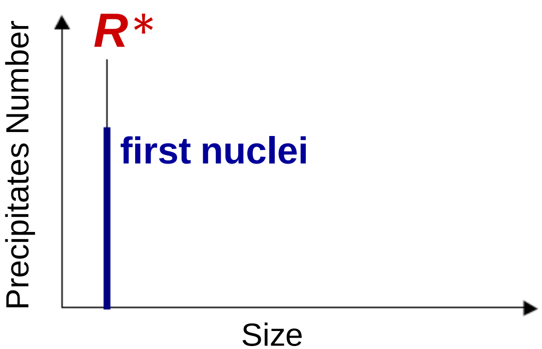
volume energy
(spherical shape assumed)

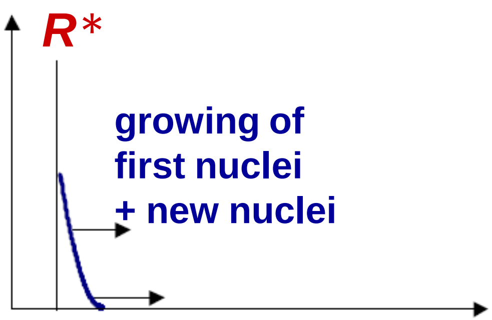
surface energy
($\gamma \approx 0.5$ mJ/m², no stress effect)

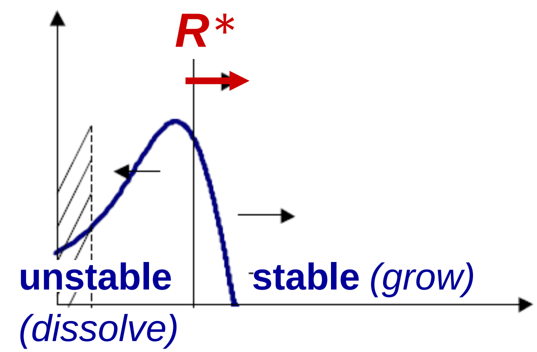


critical radius *R**

nucleation enthalpy barrier ΔG*







[D.A. PORTER, K.E. EASTERLING, *Phase transformation in metals and alloys*, London: Chapman & Hall, (1992), 514p]
[R. WAGNER, R. KAMPMANN, *Materials science and technology: a comprehensive treatment*, vol. 5, p. 213-302: *Homogeneous second phase precipitation*, John Wiley & Sons Inc., (1991)]

THERMODYNAMICAL MODELING of the PRECIPITATION

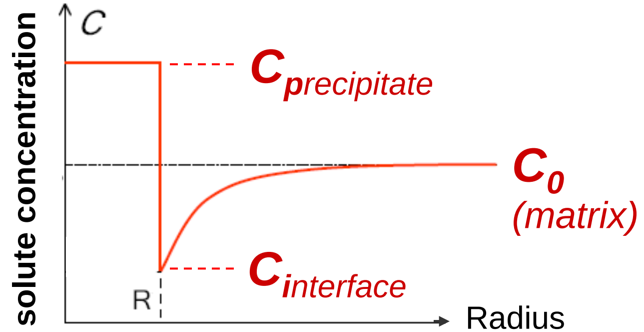
CLASSICAL NUCLEATION THEORY

2) GROWTH (driving force: *diffusion*)

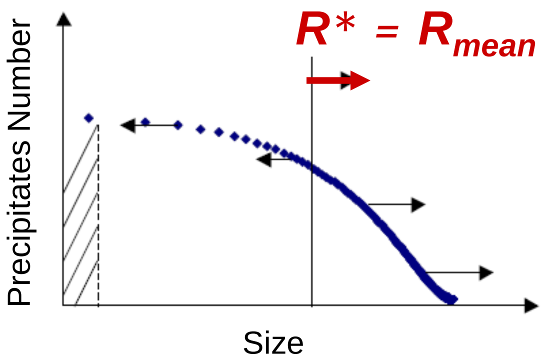
$$\underbrace{\frac{dR}{dt}}_{\text{growth rate}} = \frac{D}{R}\frac{C^0 - C^i}{C^p - C^i}$$

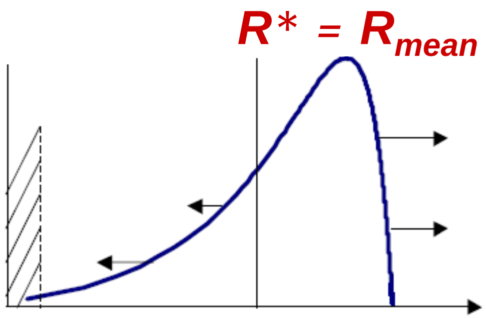
diffusion coefficient

from ***Fick's law***
(spherical coordinates)



concentration near the precipitate/matrix interface





same **(distribution density)**

Further refinements:

- **Gibbs-Thomson effect**
- **non stoechiometric binary alloyed precipitates**

3) OSTWALD coarsening (consequence of the *Gibbs-Thomson* effect)

$$\overline{R}^3(t) - \overline{R}^3_{mean}(t) \propto t$$

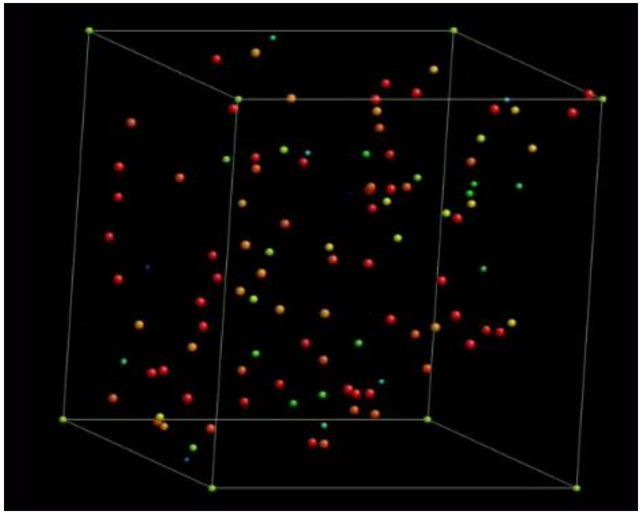
(LSW theory**)**

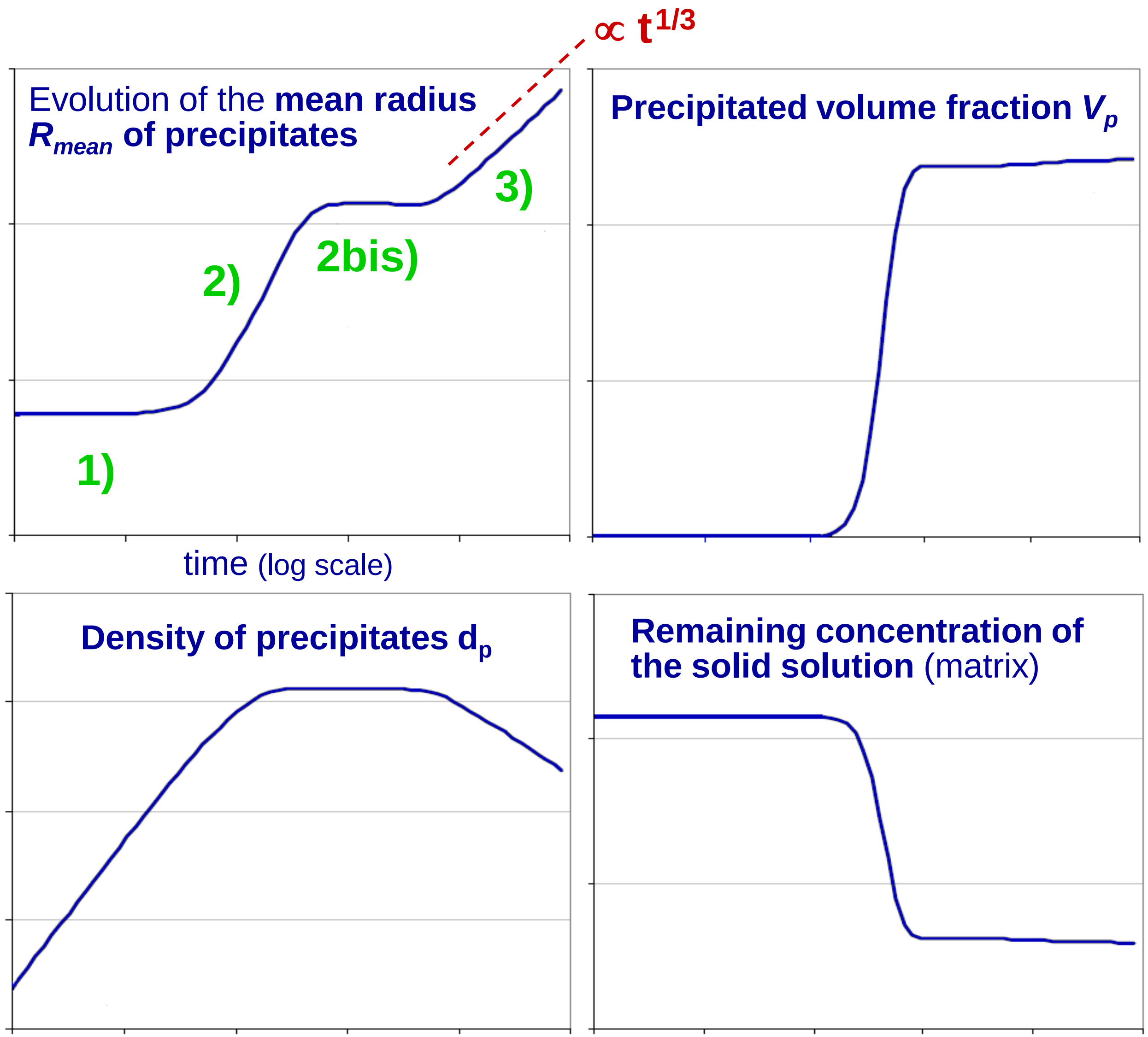
[I.M. LIFCHITZ, V.V. SLYOSOV, *J. Phys. Chem. Solids*, **19**, 1/2, (1961), 35-50]

[C. WAGNER, *Z. Electrochem*, **65**, (1961), 581]

THERMODYNAMICAL MODELING of the PRECIPITATION

CLASSICAL NUCLEATION THEORY *(summary)*





1) NUCLEATION

2) GROWTH

2bis) shrinkage of *smallest* precipitates

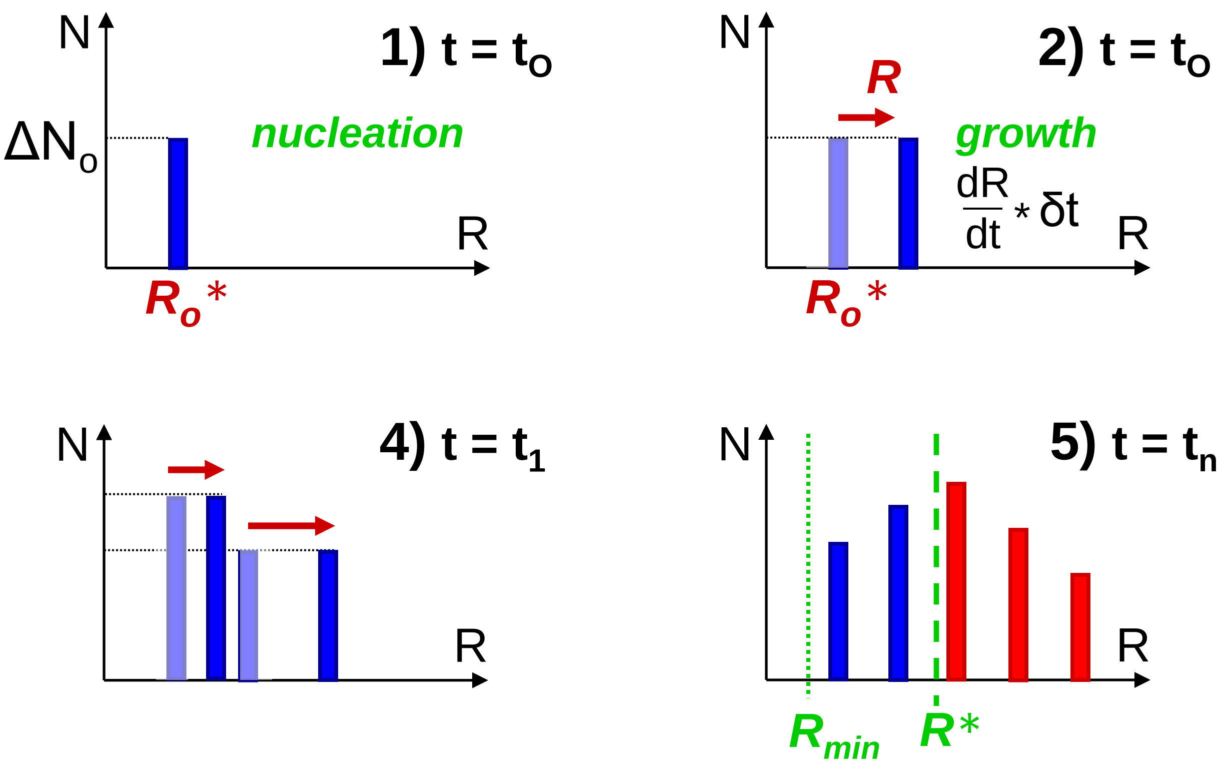
3) Ostwald coarsening

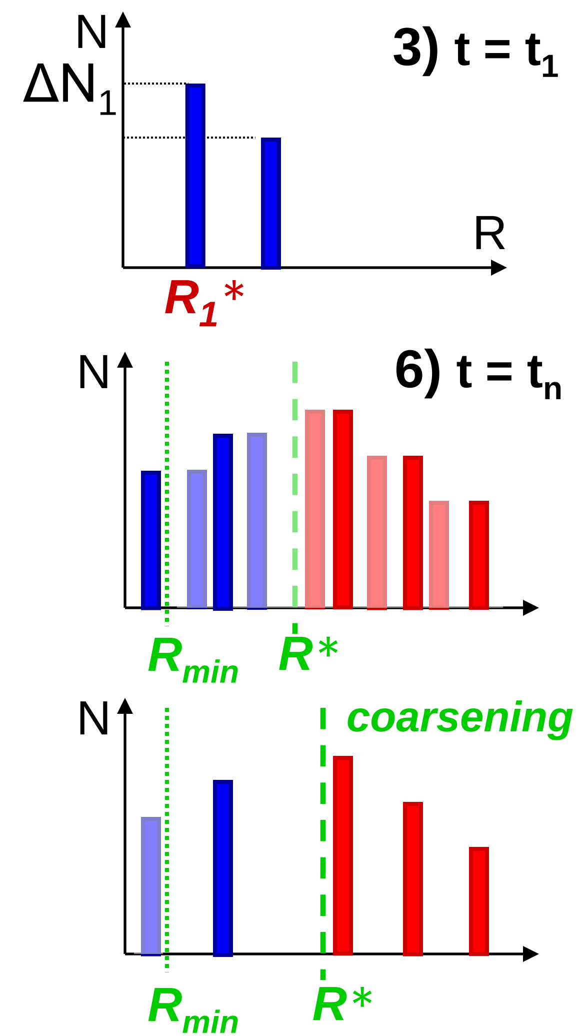
THERMODYNAMICAL MODELING of the PRECIPITATION

NUMERICAL CALCULATIONS *(multi-class approach)*

$$\frac{dN}{dt} = N_0 \cdot \beta^* \cdot Z \cdot \exp\left(-\frac{\Delta G^*}{kT}\right) \cdot \left[1 - \exp\left(-\frac{t}{\tau}\right)\right]$$

$$\frac{dR}{dt} = \frac{D}{R} \cdot \left[\frac{C'^{\alpha} - C'^{\alpha}_i}{C'_p - C'^{\alpha}_i}\right]$$





[D. ACEVEDO, *PhD thesis*, (2007), INSA-Lyon]

[D. ACEVEDO, M. PEREZ, *Computat. Materials*, (2009)]

THERMODYNAMICAL MODELING of the PRECIPITATION

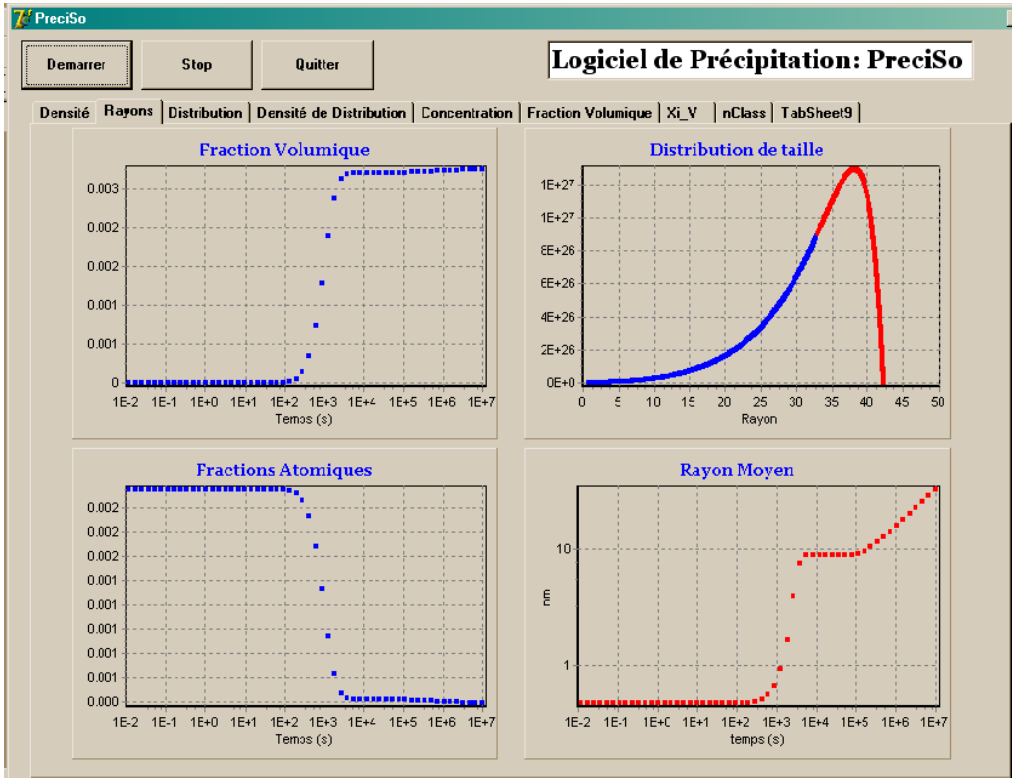
- **Precipitation in the FeNbCN system**

[M. PEREZ, E. COURTOIS, D. ACEVEDO, T. EPICIER, P. MAUGIS, *Phil. Mag. Letters*, **87**, (2007), 645-656]

- **Reversion in the FeVC and FeVNbC systems**

[D. ACEVEDO, *PhD thesis*, (2007), INSA-Lyon]

[D. ACEVEDO, M. PEREZ, T. EPICIER, E. KOZESCHNICK, F. PERRARD, T. SOURMAIL, p.987-999 in "New developments on Metallurgy and Applications of High Strength Steels", Vols 1/2, *Min., Metals & Mater. Soc.*, Warrendale – USA, (2009)]



- **Reversion and grain growth control in the FeCVNbN system**

[C. LEGUEN, *PhD thesis*, (2010), INSA-Lyon]

Dissolution in the FeVC system

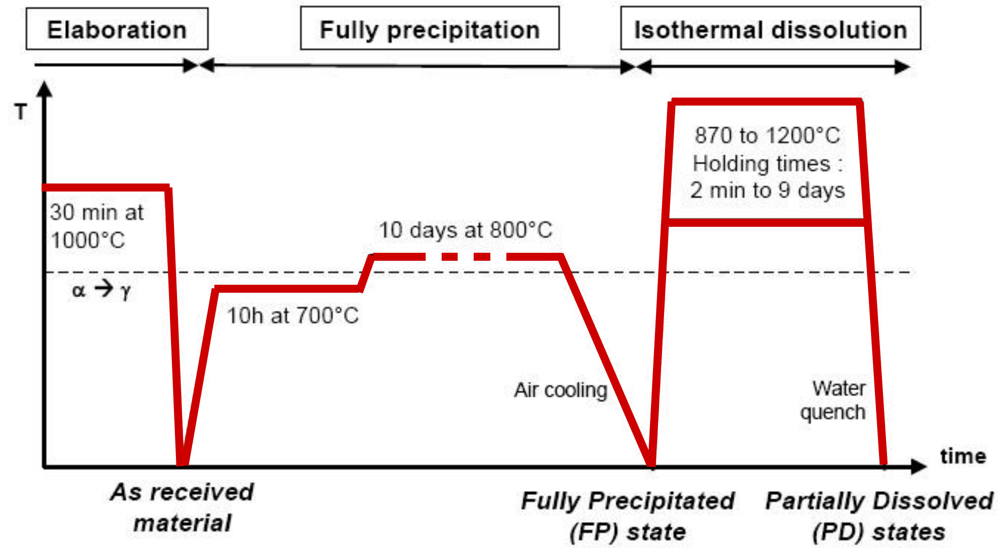
ASCOMETAL

[D. ACEVEDO, PhD thesis, INSA-Lyon, (2005)]

model alloy

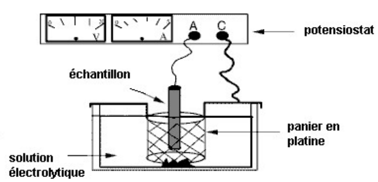
Fe - V 0.2 wt. %, C 0.48 %

- **STUDY of the DISSOLUTION of Vanadium Carbides during REVERSION treatments**



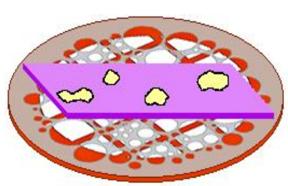
- **EXPERIMENTAL techniques**

- Fraction of precipitated elements



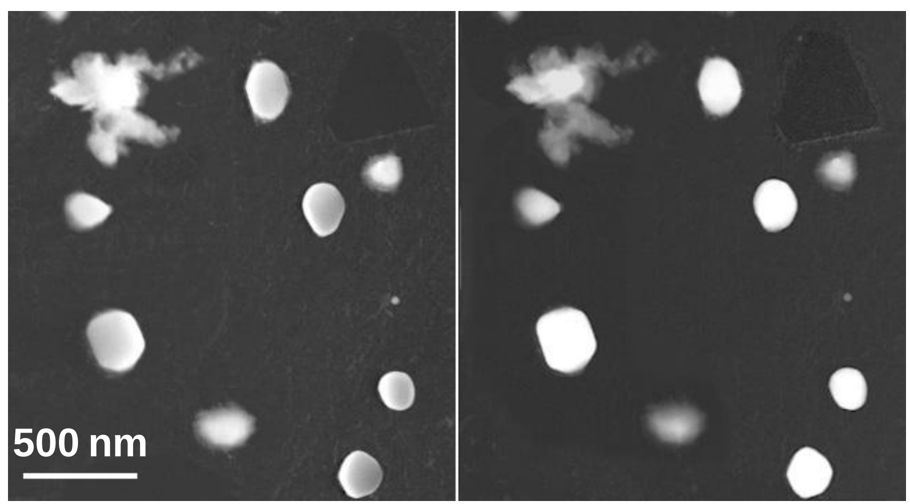
Electrolytic dissolution + **ICP** (**I**nductive **C**oupled **P**lasma) spectroscopy

- Precipitates statistics



Extraction replicas
(+ thin foils)

STEM in SEM

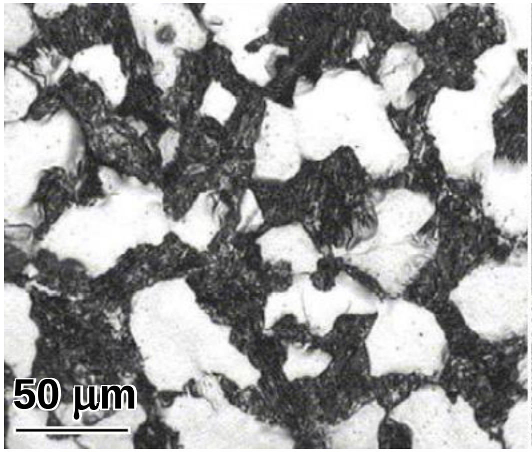


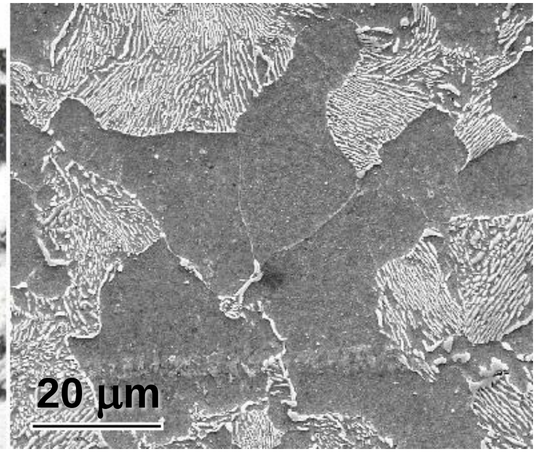
HAADF in TEM

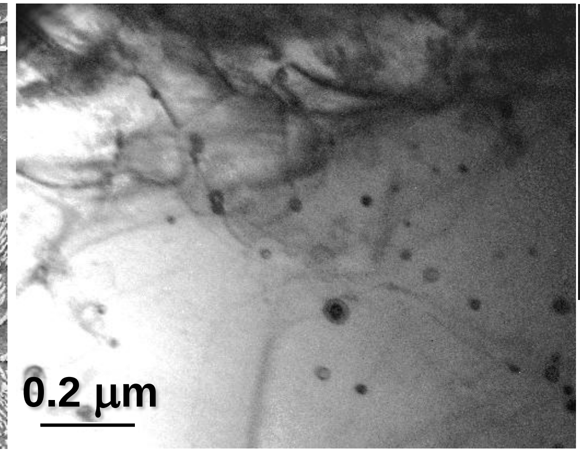
[ACEVEDO-REYES D., PEREZ M., VERDU C., BOGNER A., EPICIER T., *J. of Microscopy*, **232**, 1, (2008), 112–122]

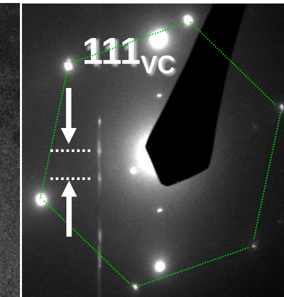
ASCOMETAL

Fully Precipitated state (ferrito-perlitic)







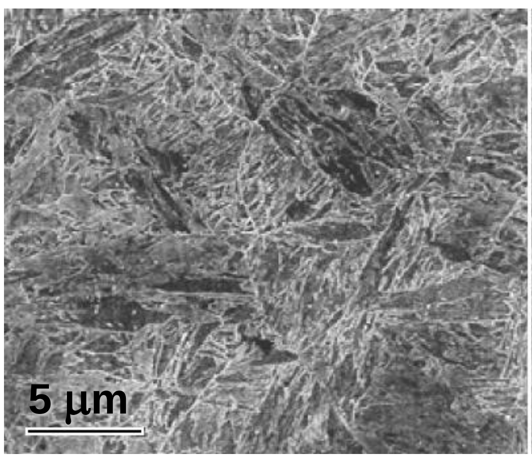


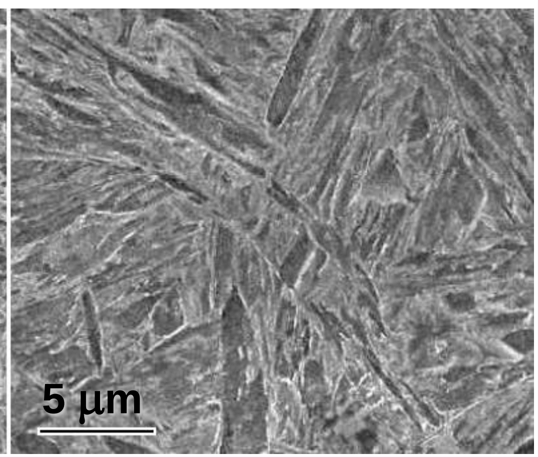
martensite

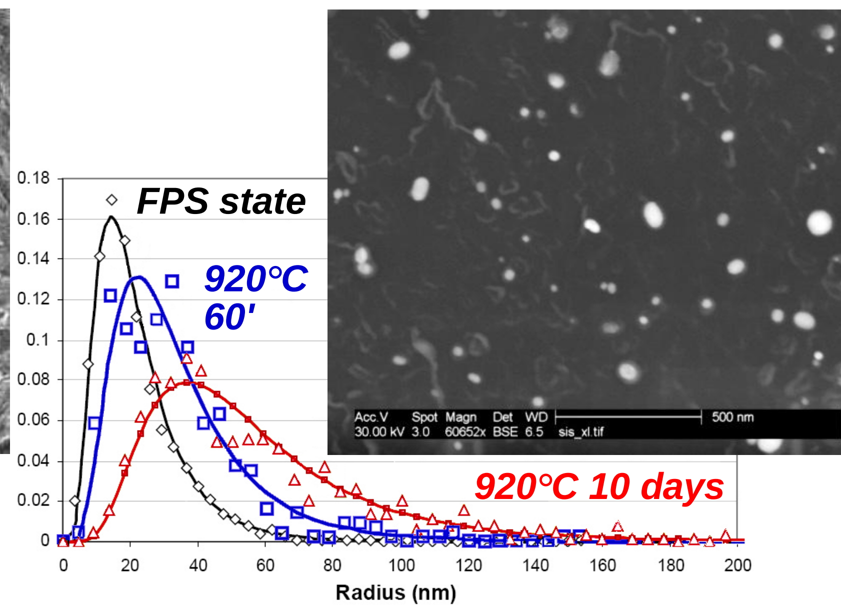
ordered V_6C_5

[T. EPICIER et al., *Phil. Mag.*, **88**, 1, (2008), 31-45]

Reversion states (martensitic)

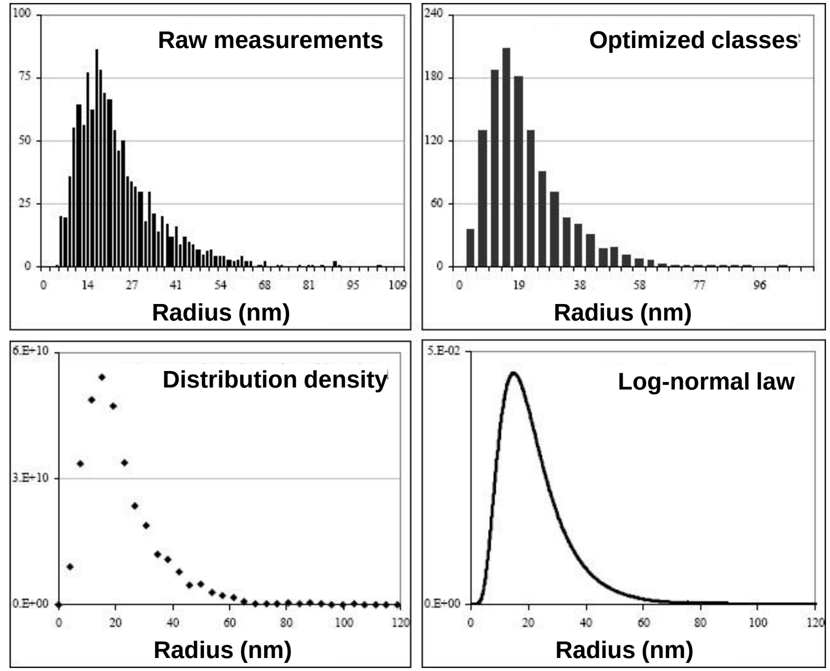






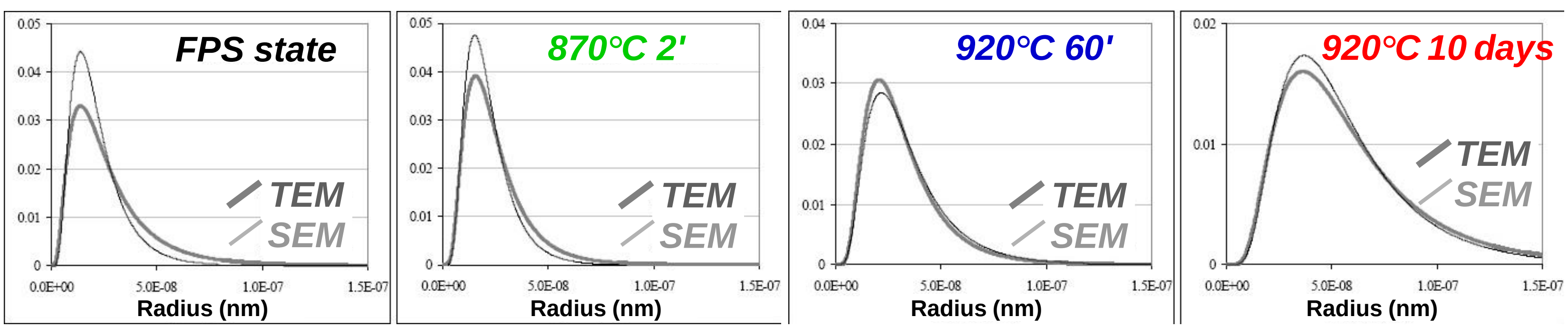
• Rk.1: normalizing histograms

FPS state



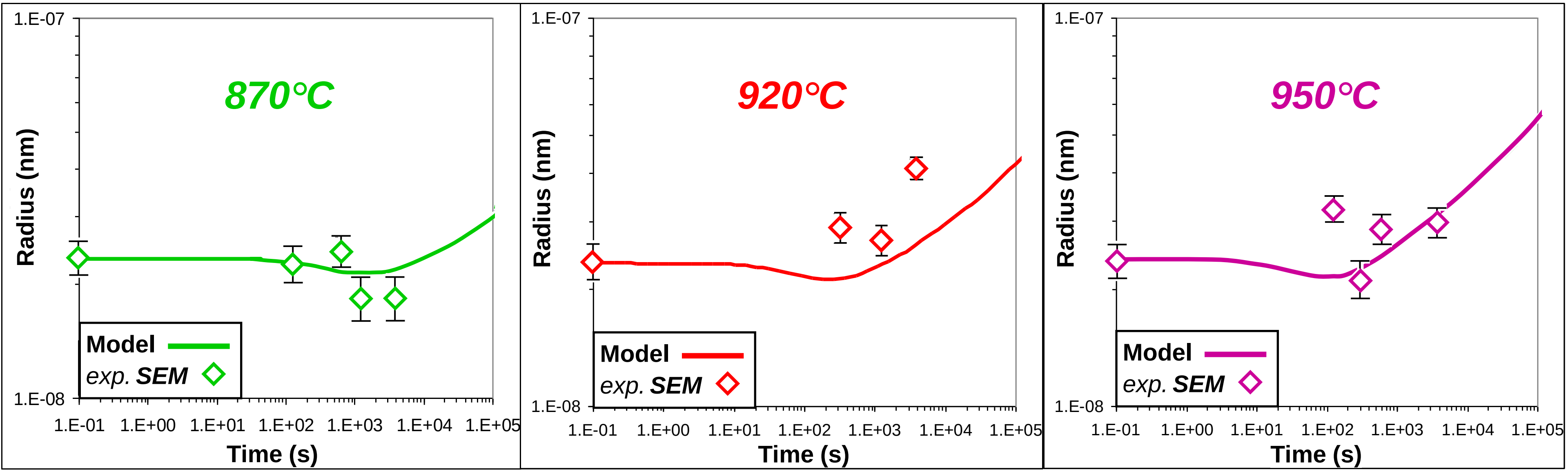
t (min)	870°C	920°C	950°C
0	1227	1227	1227
2	920		394
5		114	606
10	308		356
20	302	533	
60	1074	426	345

• Rk.2: SEM-TEM correspondence

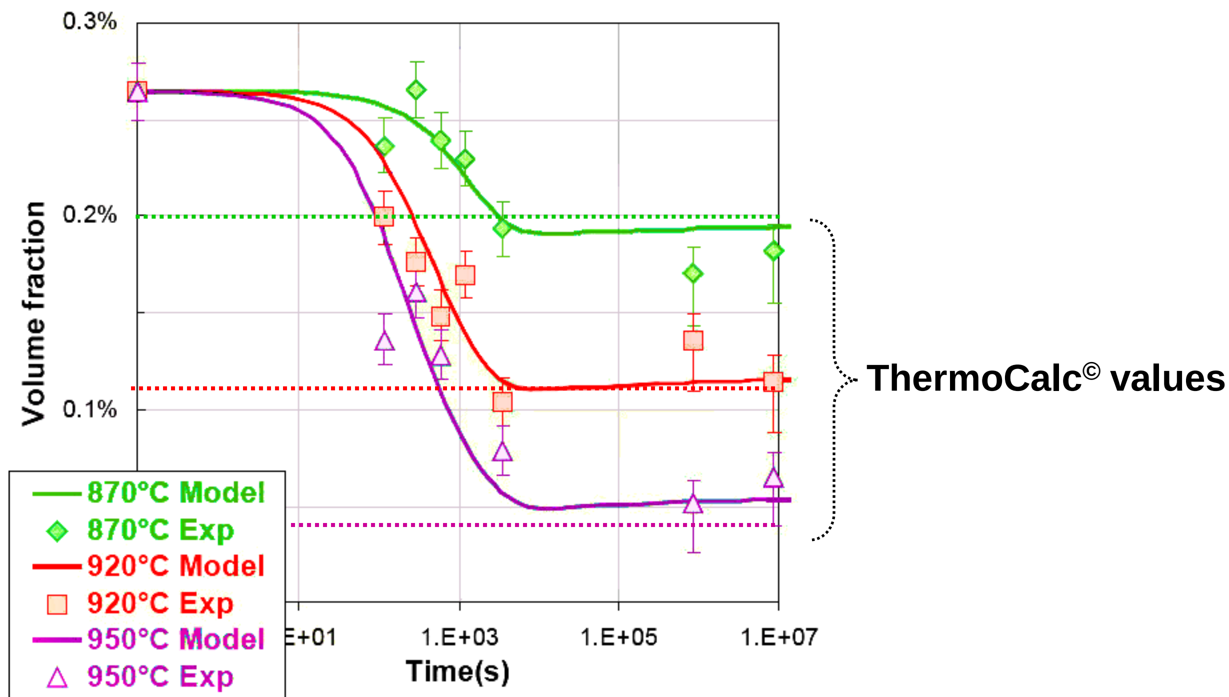


- **Size of precipitates**

ASCOMETAL

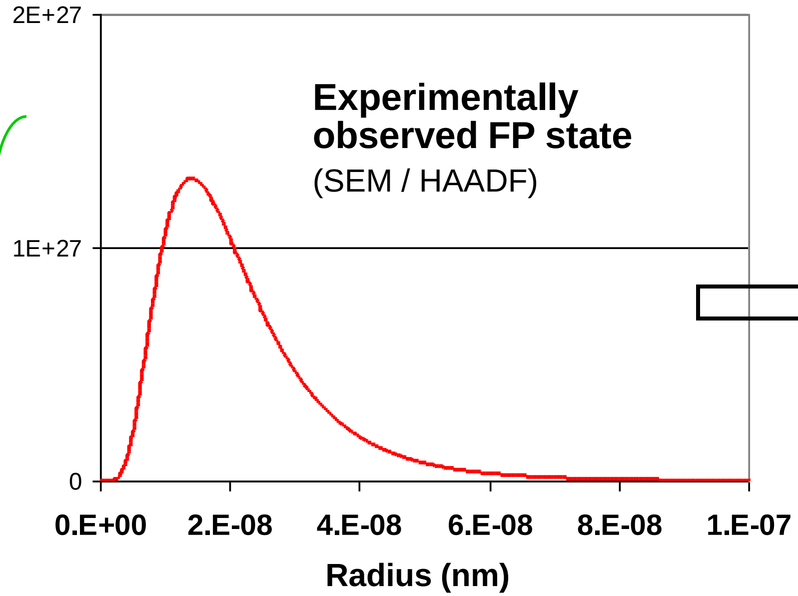


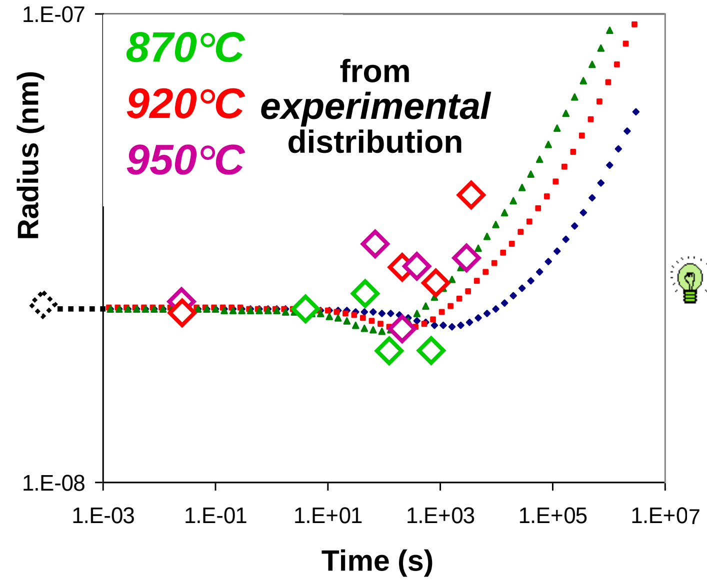
- **Volume fraction of precipitates**



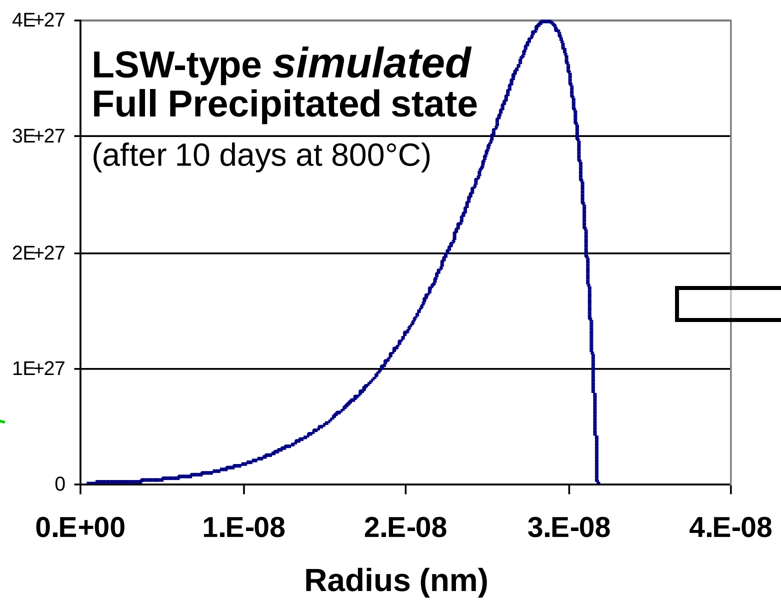
• Consistency of results: *effect of the 'starting' precipitate distribution*

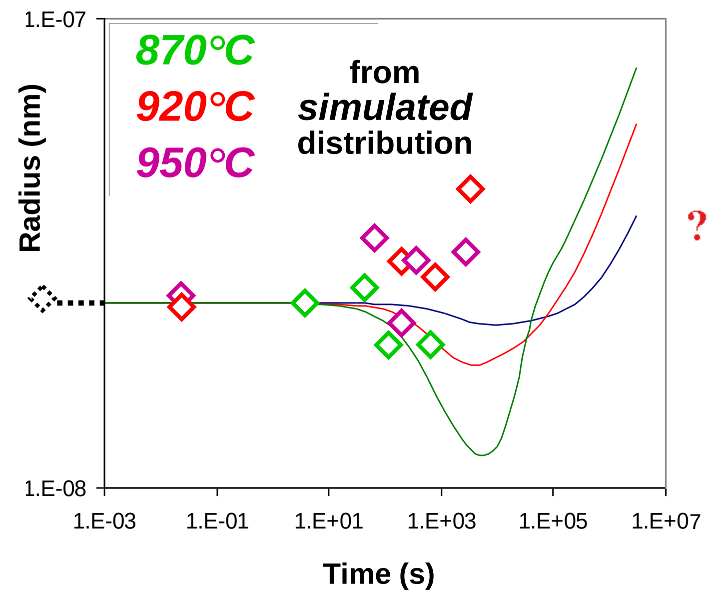
ASCOMETAL





same mean radius

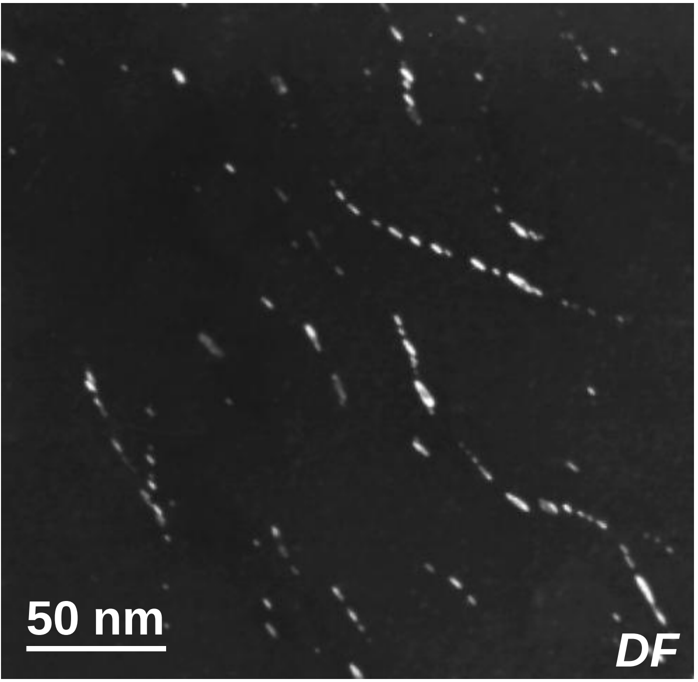




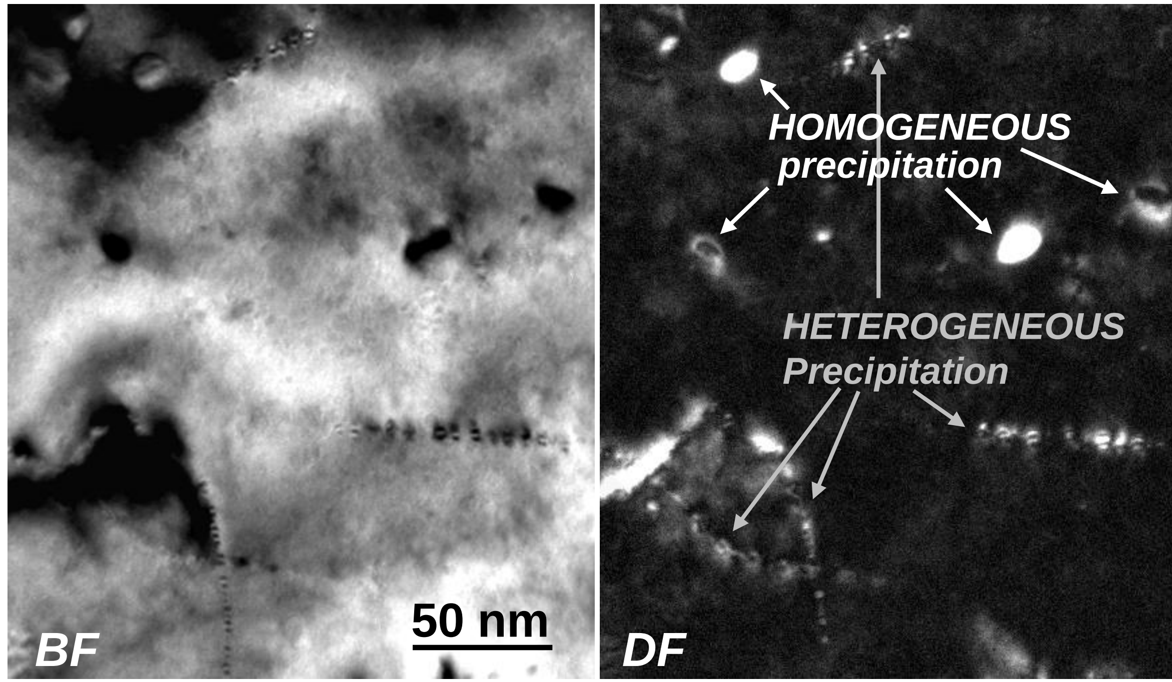
Precipitation in the FeNbCN system

model steel
Fe - Nb 790 wt. ppm, C 120 ppm, N 10 ppm
(800°C, 30')

Model steel
Fe - Nb 843 ppm, C 59 ppm, N 64 ppm
(650°C, 30')



heterogeneous precipitation of NbC (c.f.c.)
in α-Fe
'BAKER-NUTTING' O.R.



heterogeneous precipitation of NbC (c.f.c.)
and homogeneous precipitation of NbN (c.f.c.)
in α-Fe
('BAKER-NUTTING' O.R.)

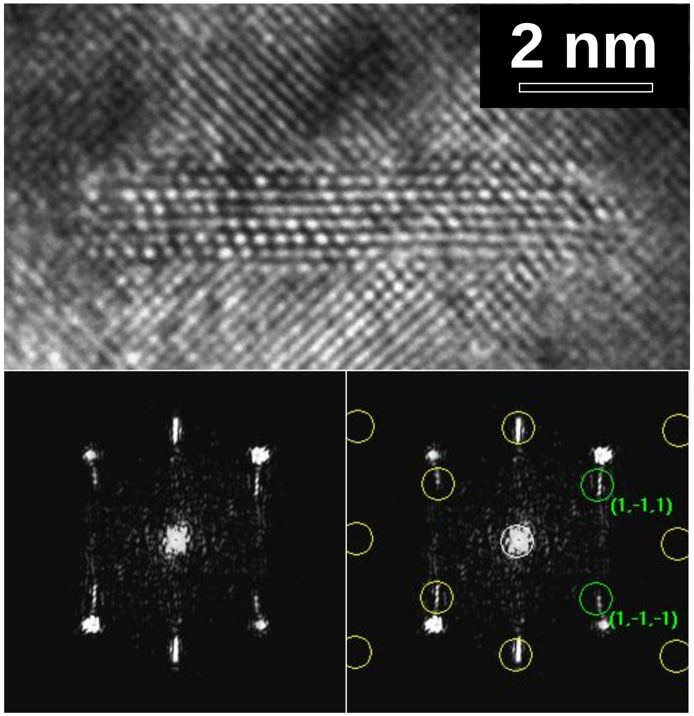
[É. COURTOIS, T. EPICIER, C. SCOTT, *Micron*, **37** (2006), 492-502]

[T. EPICIER, *Adv. Eng. Mater.* **8**, 12, (2007), 1197-1201]

model steel

Fe - Nb 790 wt. ppm, C 120 ppm, N 10 ppm

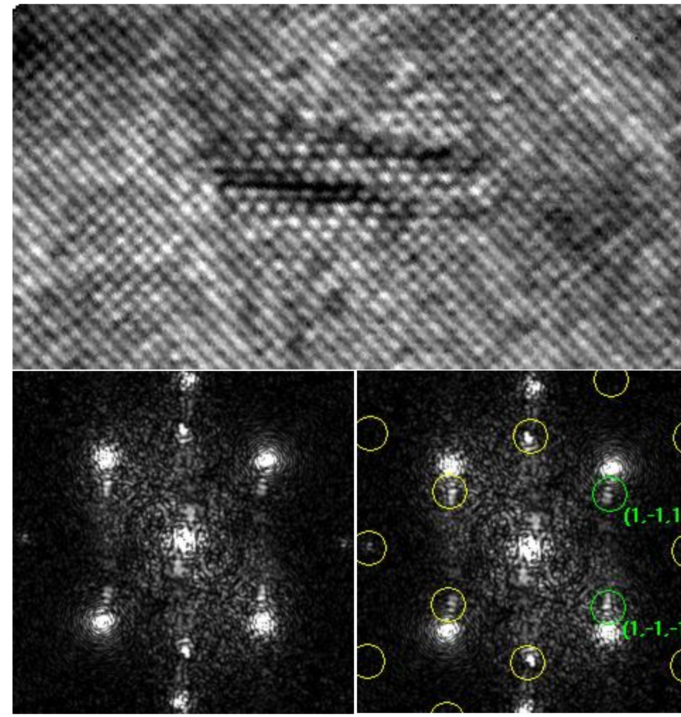
(800°C, 30')



Model steel

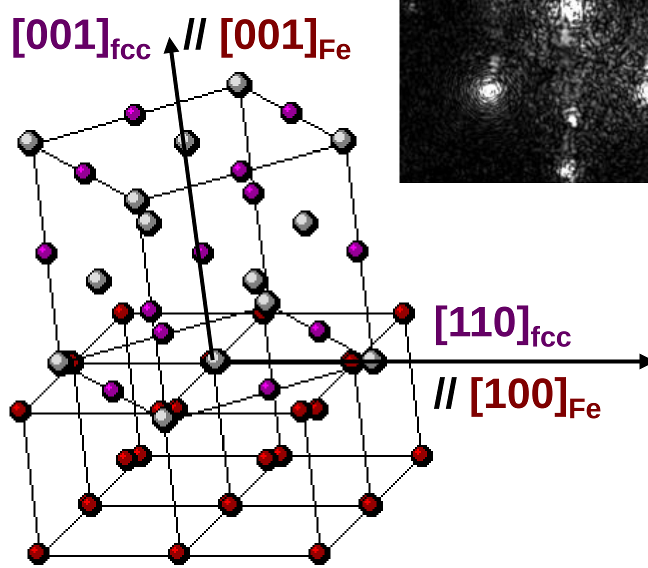
Fe - Nb 843 ppm, C 59 ppm, N 64 ppm

(650°C, 30')



BAKER-NUTTING O.R.:

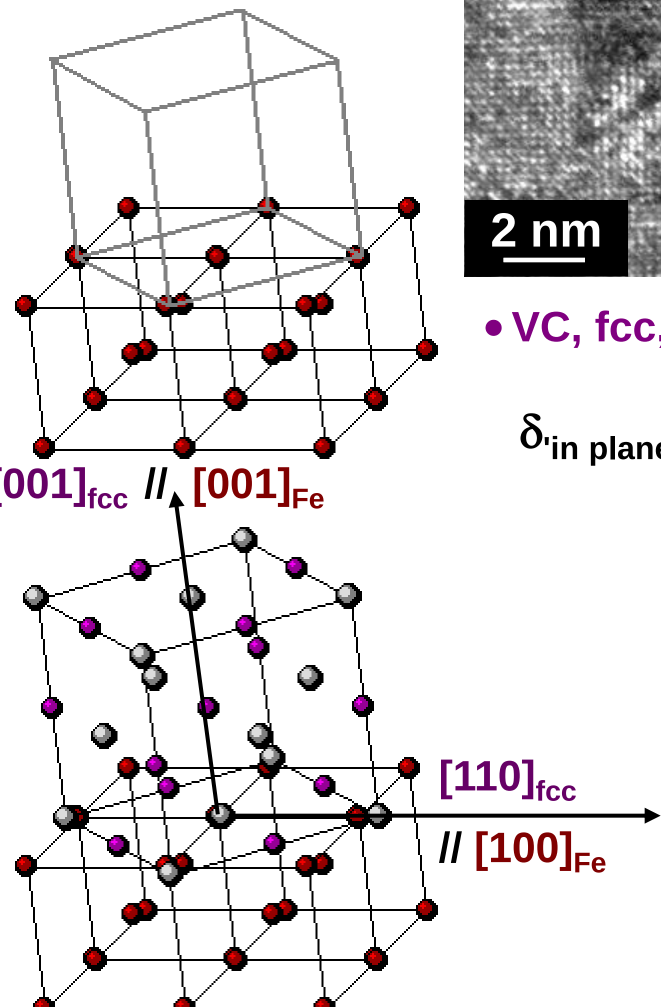
$[110]_{F.C.C.}$ // $[001]_{Fe-\alpha}$

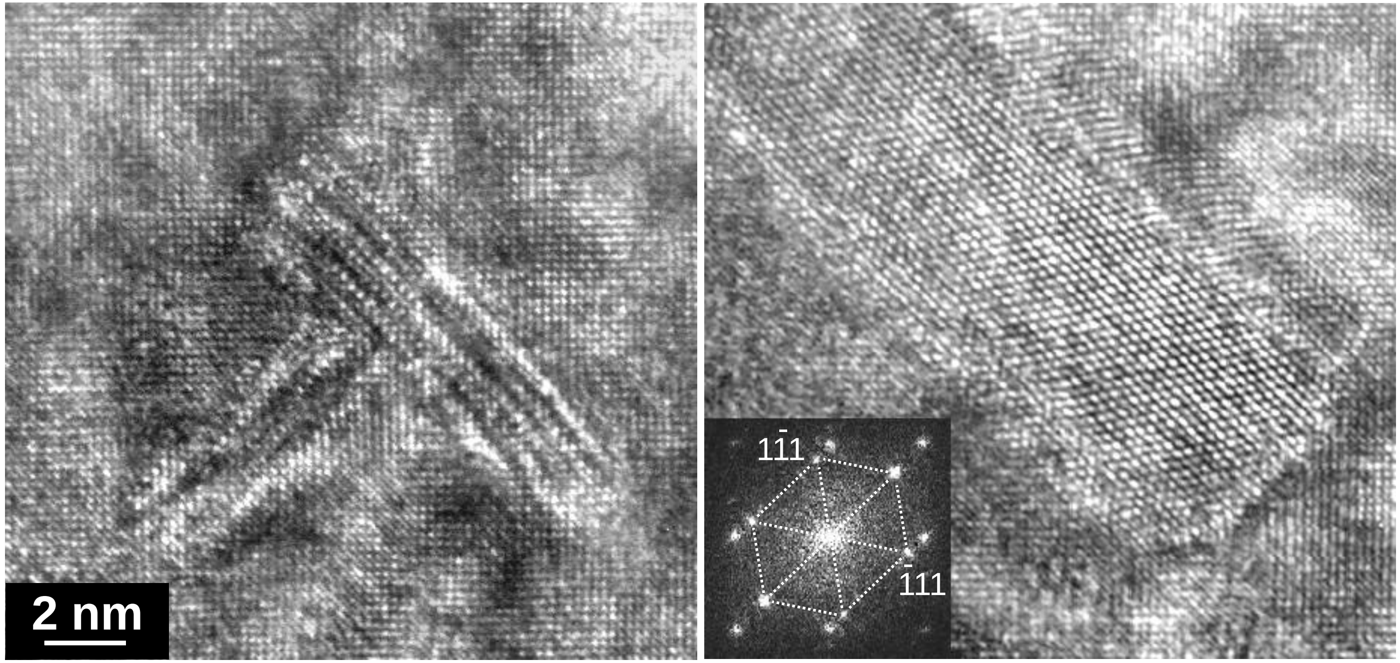


α-Fe (ferrite):
cc, a = 2.86_6 Å

- **Perfect fcc structure for *B.N.*:**

  $a_{fcc} = a_{Fe}\sqrt{2} \approx 4.05_3$ Å





- **VC, fcc, a_{VC} = 4.17 Å**

$$\delta_{'in\ plane'} = \frac{(d_{220}^{VC} - d_{100}^{Fe})}{d_{100}^{Fe}} = 2.88\ \%$$

[T. EPICIER et al., *Phil. Mag.*, **88**, 1, (2008), 31-45]

- **NbC, fcc, a_{NbC} = 4.47 Å**

$$\delta_{'in\ plane'} = \frac{(d_{220}^{NbC} - d_{100}^{Fe})}{d_{100}^{Fe}} = 10.28\ \%$$

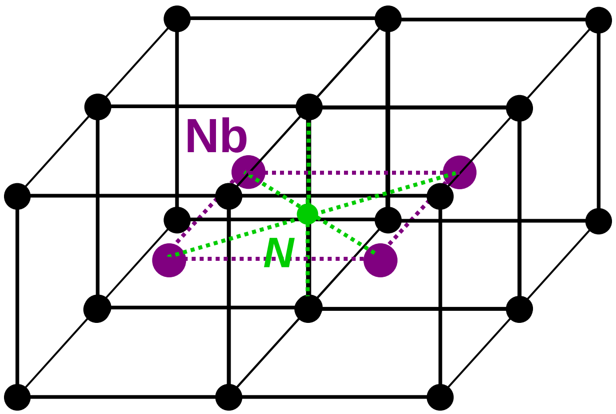
- **NbN-δ, fcc, a_{NbN} = 4.39_4 Å**

$$\delta_{'in\ plane'} = \frac{(d_{220}^{NbC} - d_{100}^{Fe})}{d_{100}^{Fe}} = 8.41\ \%$$

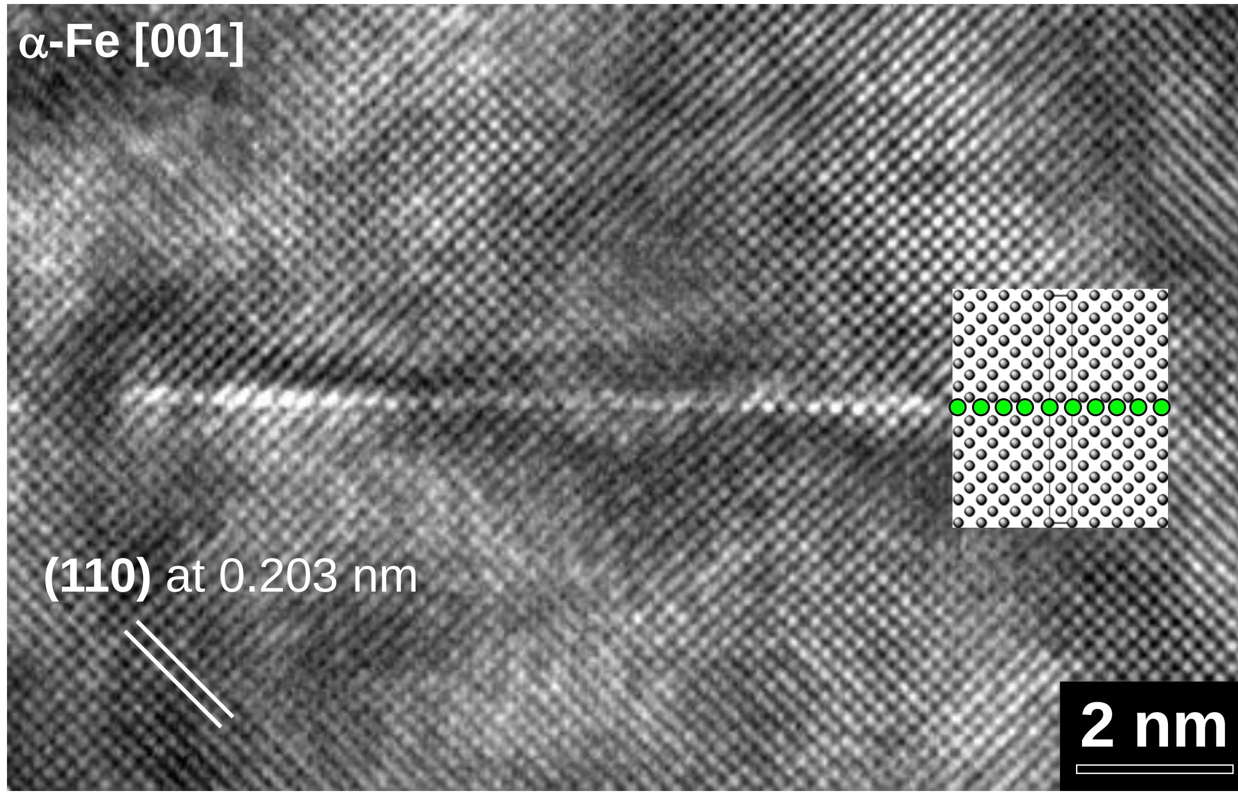
Model steel

Fe - Nb 843 ppm, C 59 ppm, N 64 ppm

(650°C, 30')



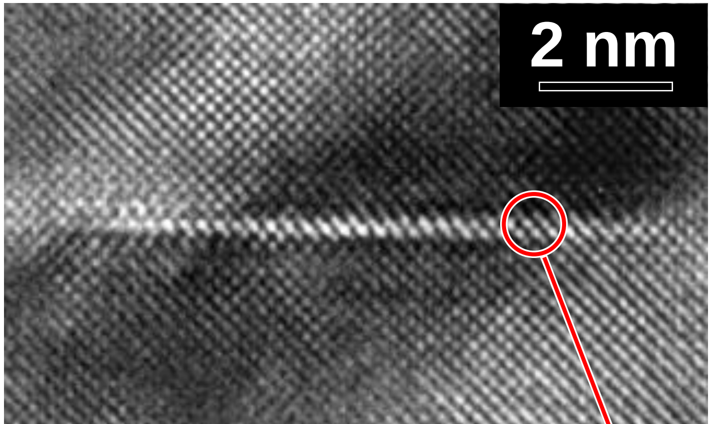
Fe ferrite



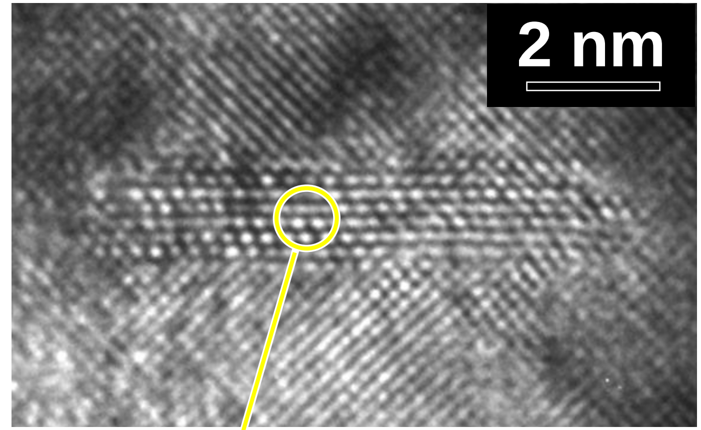
⇨ **COMPATIBLE with a Nb-(100) plane in FERRITE**

⇨ **NOT OBSERVED in the C-rich steel: NbN platelets?**

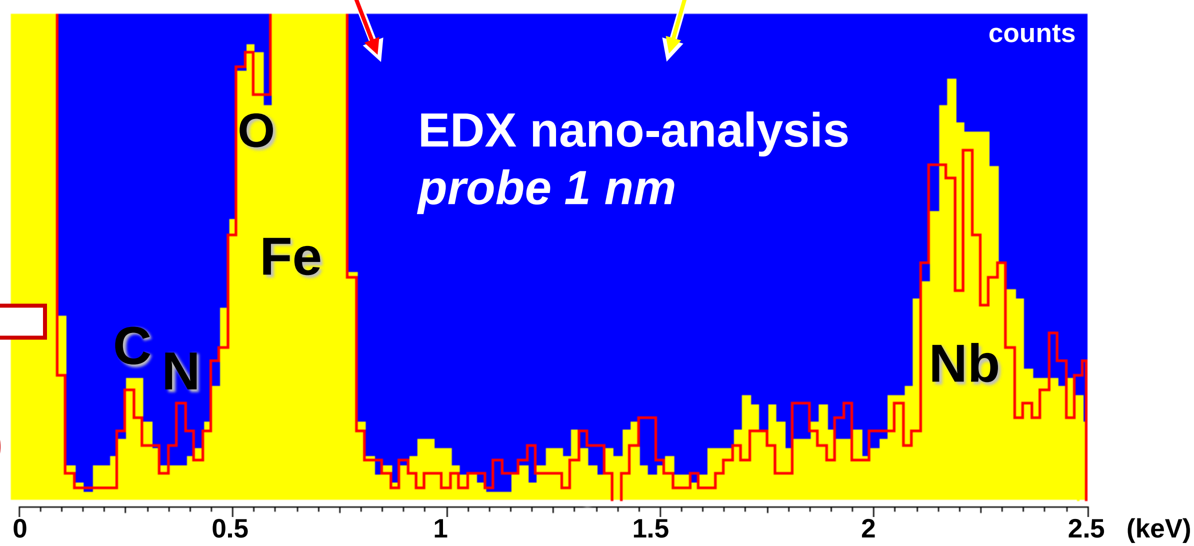
ATOMIC PLATELETS or ‘G.P. zones’



NANO-PRECIPITATES



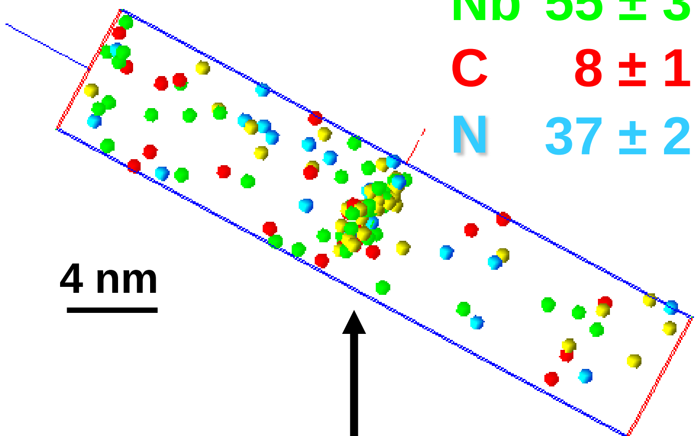
EVIDENCE for a DOUBLE POPULATION of ‘OBJECTS’



ATOMIC PLATELETS: N-enriched (?)

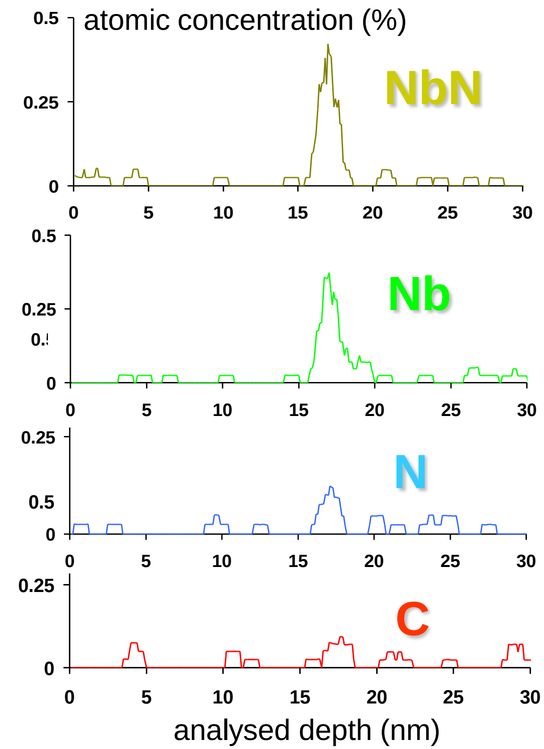
Atom Probe Tomography

[T. EPICIER, F. DANOIX, F. VURPILLOT, *PTM 2010, Avignon-F, and to be published*]

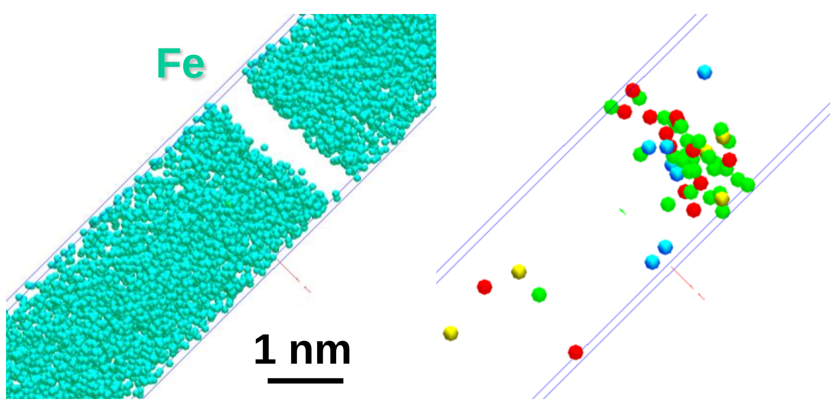


Nb 55 ± 3 at. %
C 8 ± 1 at. %
N 37 ± 2 at. %

N-enriched
(ATOMIC PLATELETS…)



CONFIRMATION of a DOUBLE POPULATION of 'OBJECTS'

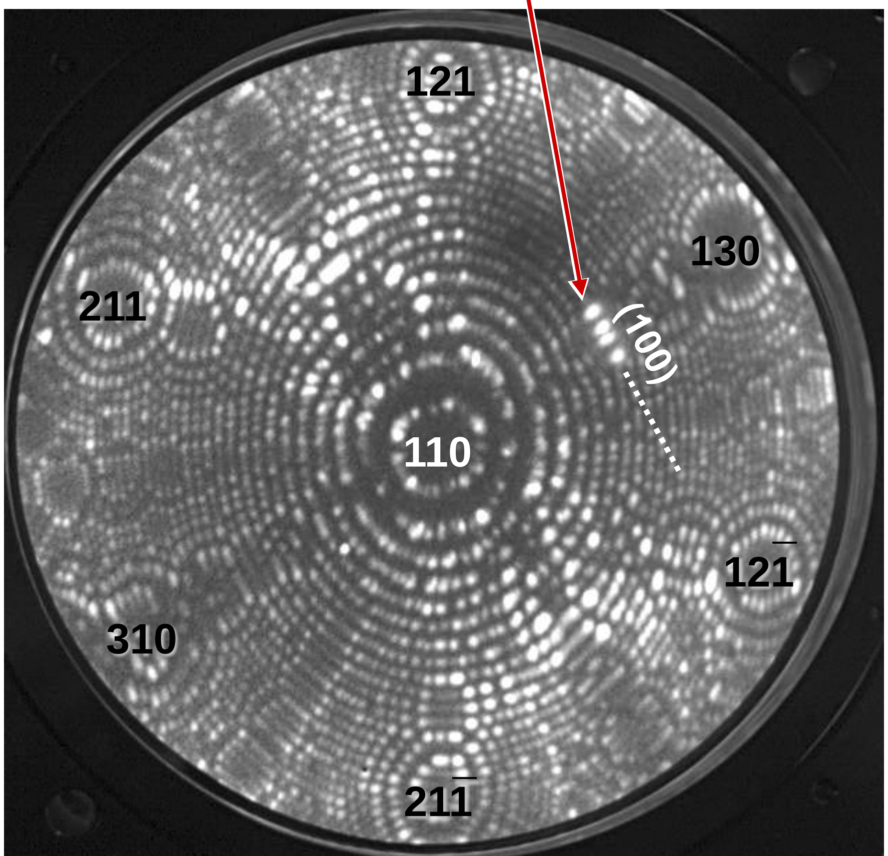


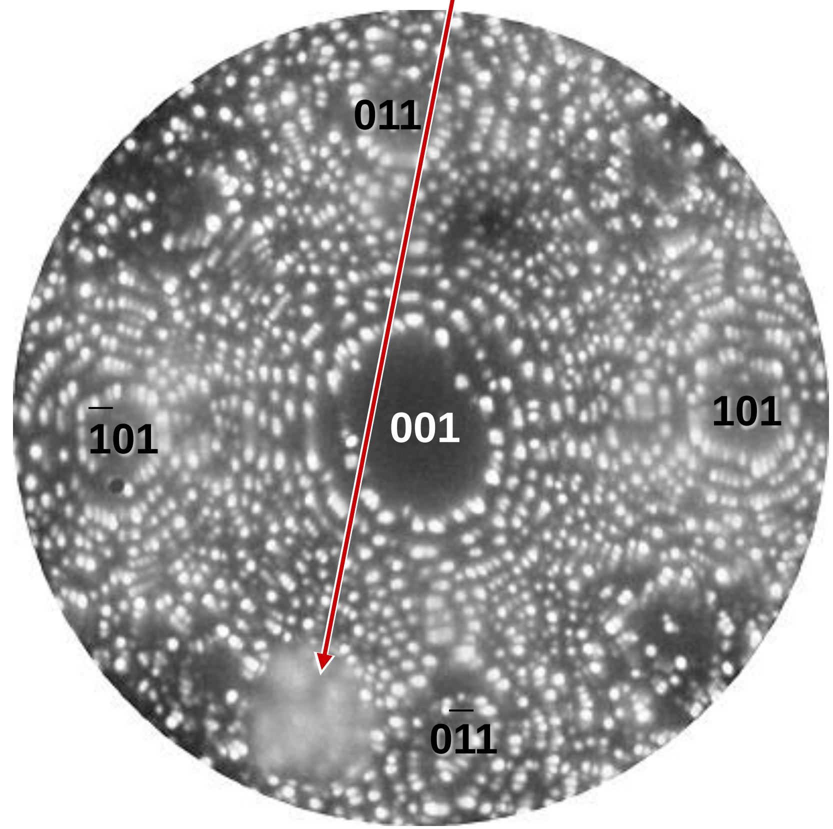
'carbo-nitrides'
(NANO-PRECIPITATES…)

Nb 47 ± 3 at. %
C 20 ± 1 at. %
N 33 ± 2 at. %

and Field Ion Microscopy...

CONFIRMATION of both
<u>ATOMIC PLATELETS</u> and <u>NANO-PRECIPITATES</u>
by FIELD ION MICROSCOPY

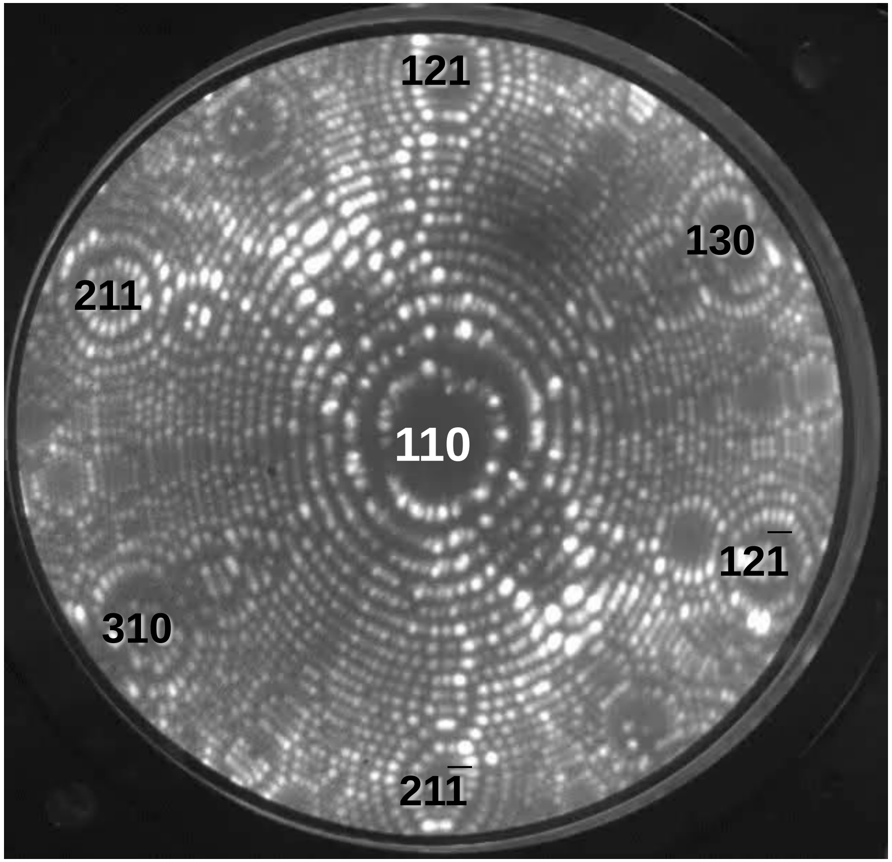




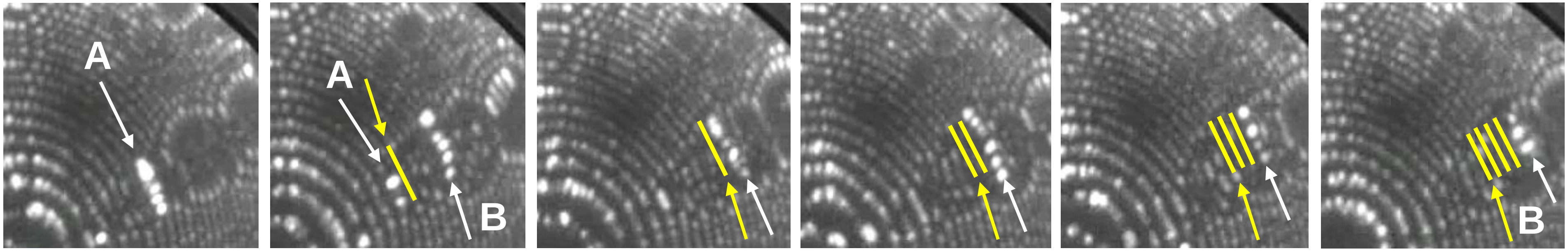
and Field Ion Microscopy...

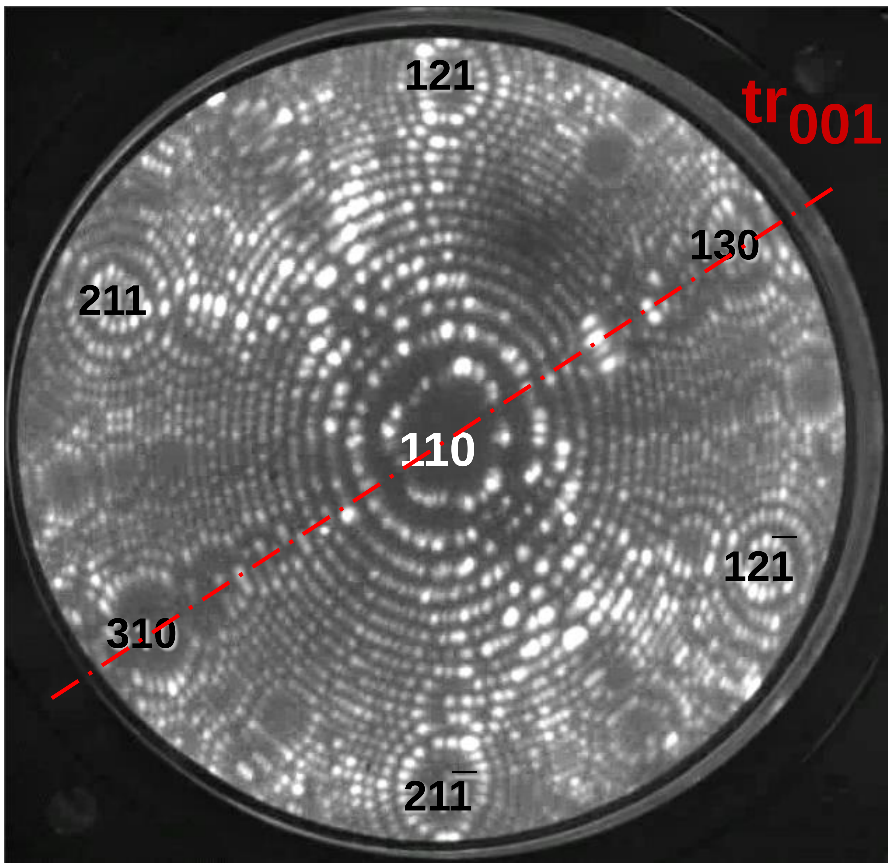
◆ **Details of the analysis of <u>ATOMIC PLATELETS</u>**

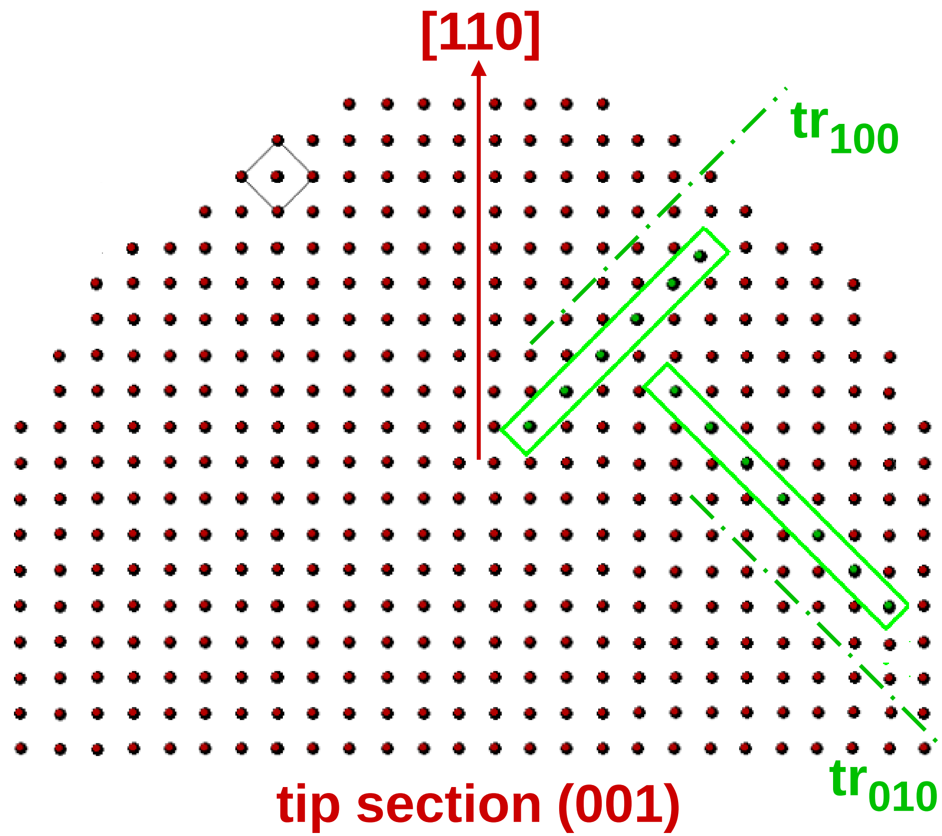
FIM evaporation sequence



and Field Ion Microscopy...

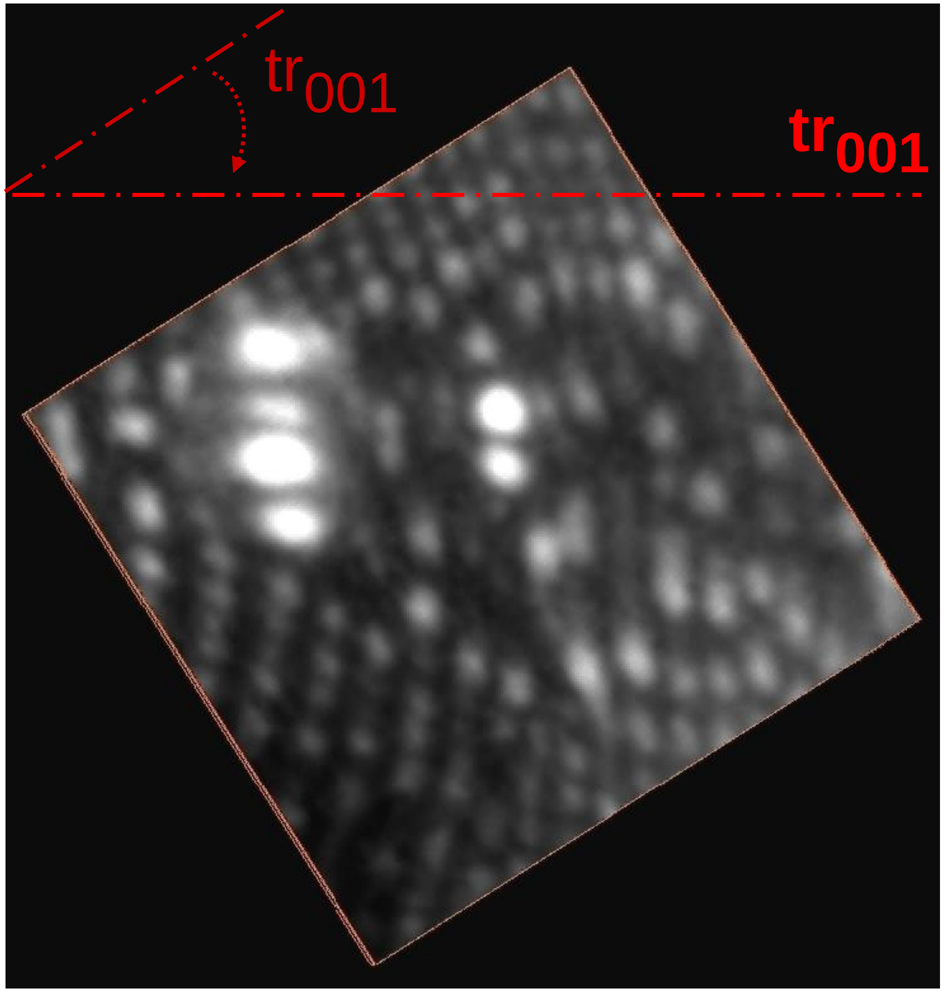


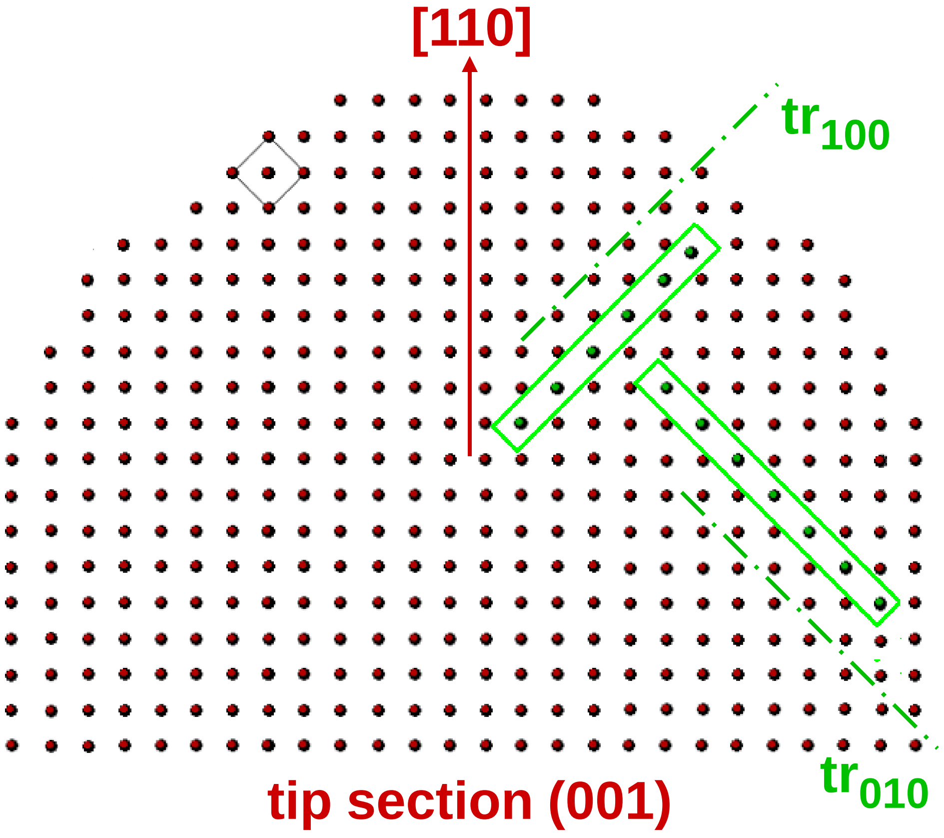




and Field Ion Microscopy...

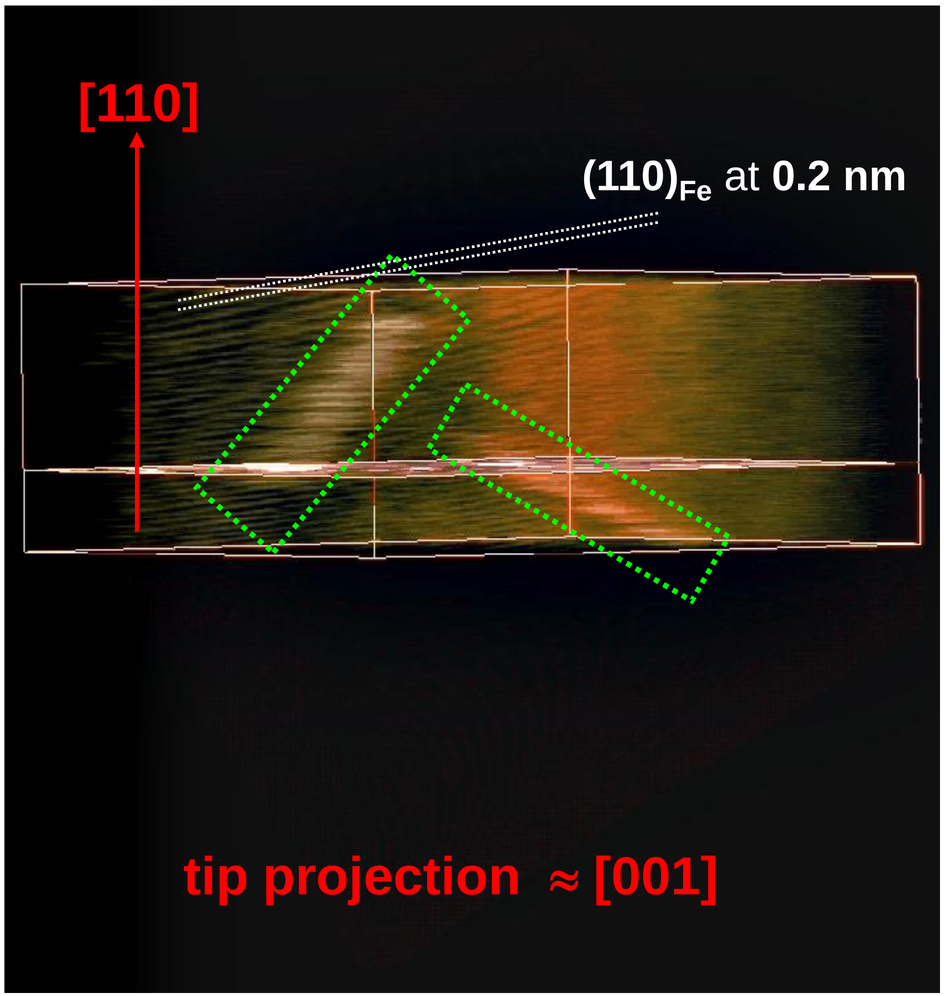
- 3D analysis of the FIM evaporation sequence

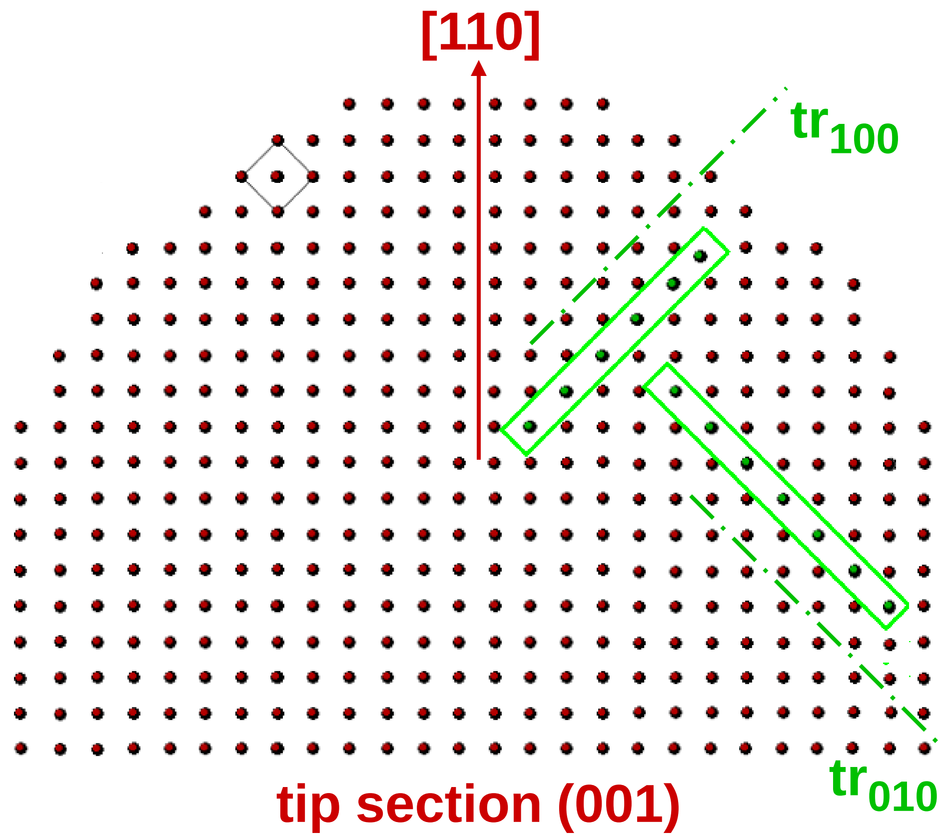




and Field Ion Microscopy...

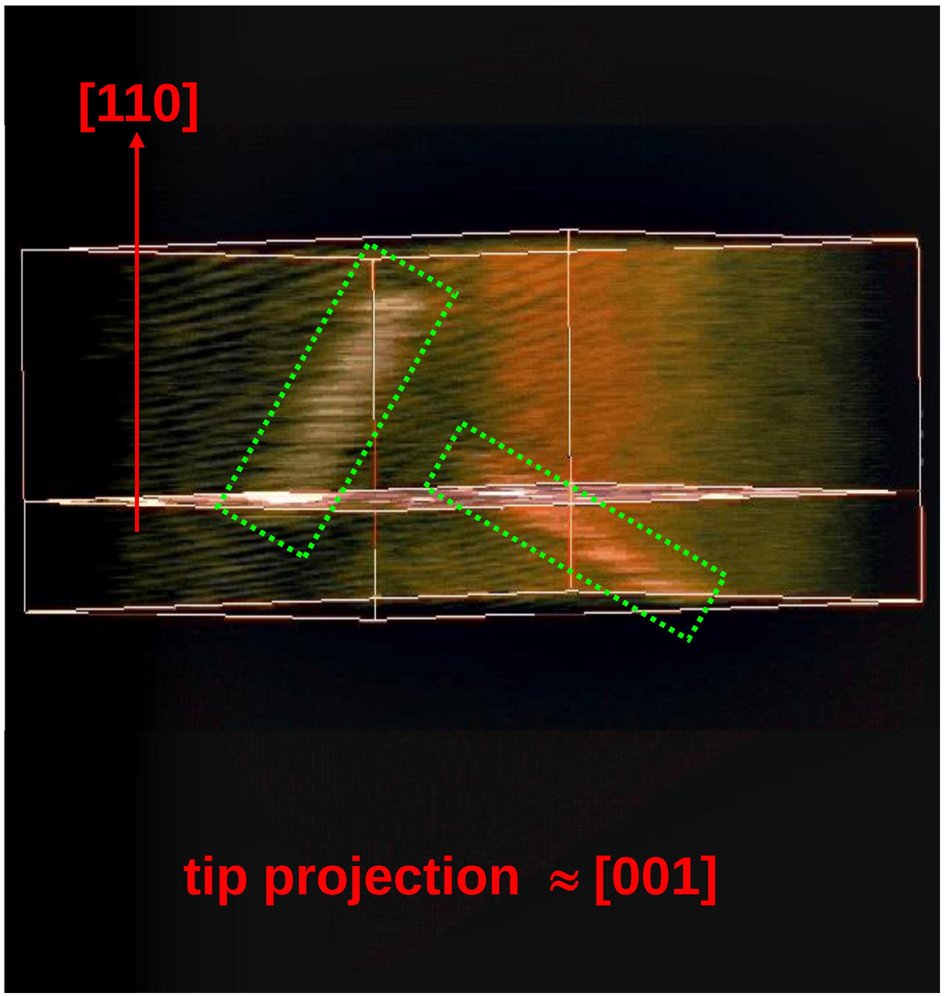
- 3D analysis of the FIM evaporation sequence

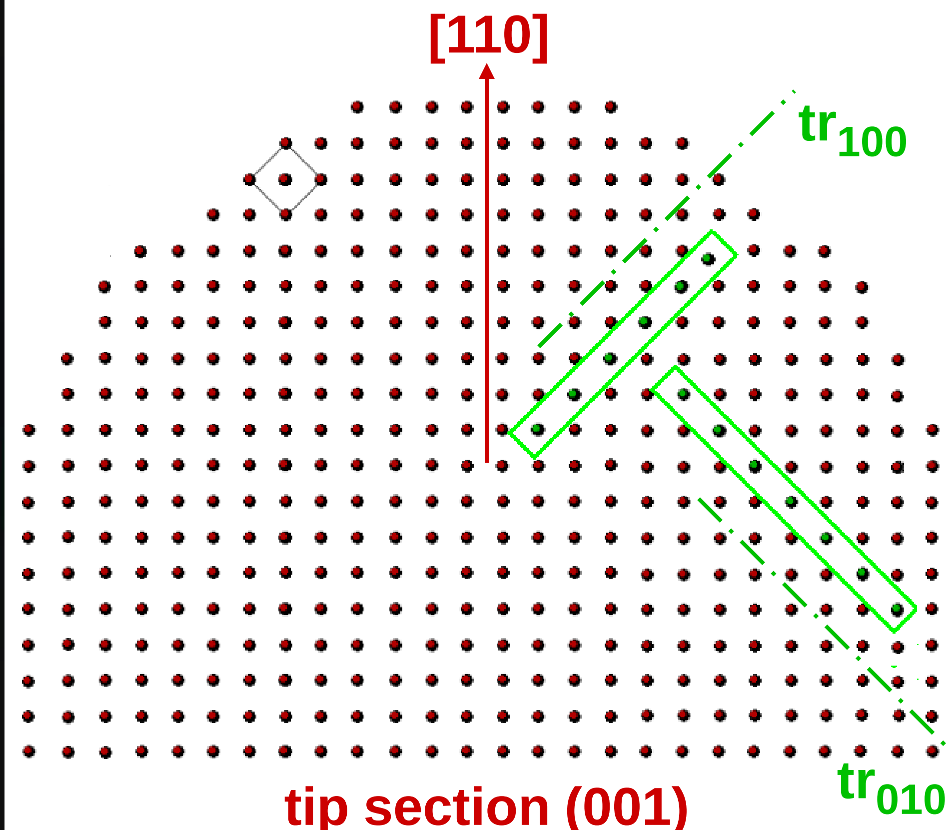




and Field Ion Microscopy...

- **3D analysis of the FIM evaporation sequence**



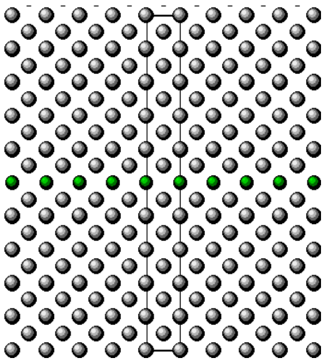


Back to High Resolution TEM...

◆ *indicative* ***HRTEM simulations***

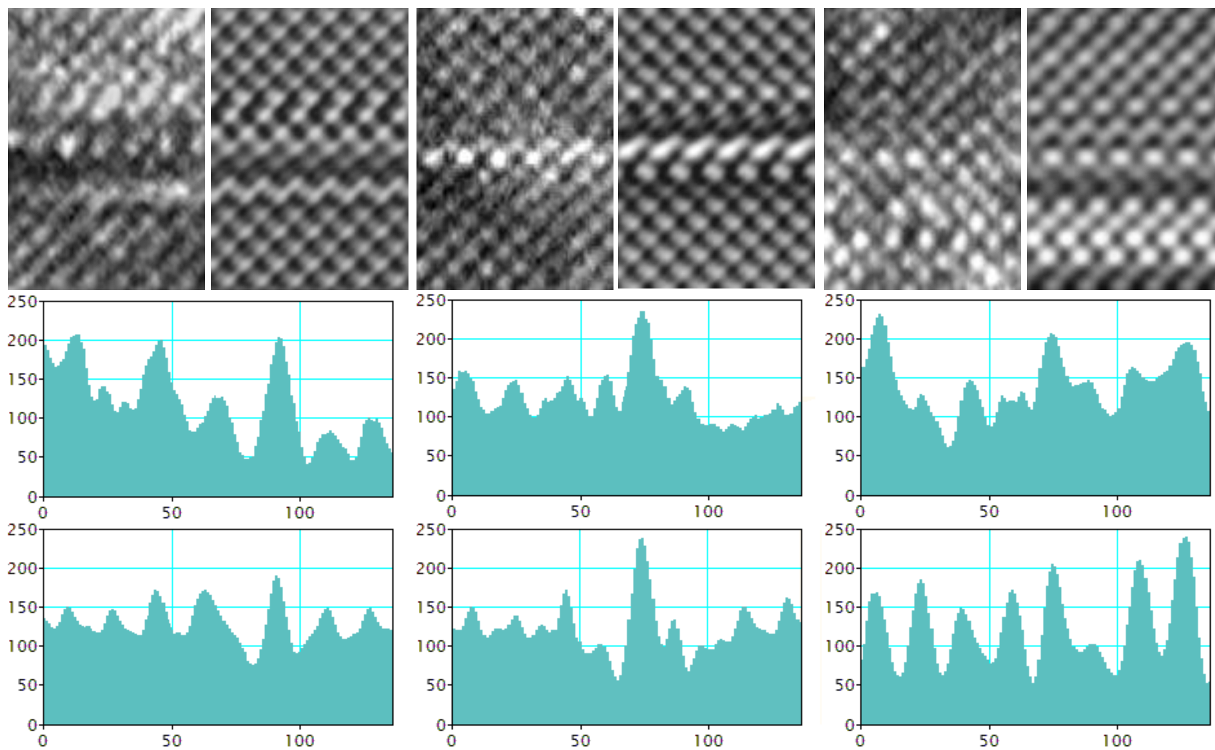
δf = - 90 nm | - 60 nm | + 45 nm

experiment / simulation | **experim^t / simulat^n** | **experim^t / simulat^n**



'rough model': substitution of Fe atoms by Nb atoms in a (001) plane

(NO metalloïd, NO atomic relaxation)



total thickness:
20 nm Fe +
20 nm platelet (Fe,Nb)

AVERAGE PROFILE ACROSS the DEFECT

experiment

simulation

⇨ **COMPATIBLE with a Nb-(100) plane in FERRITE**

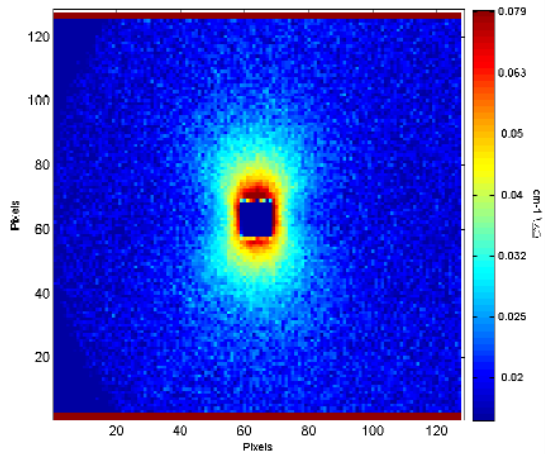
Guinier-Preston type ZONES in a pure Fe-Nb-N system

SANS study

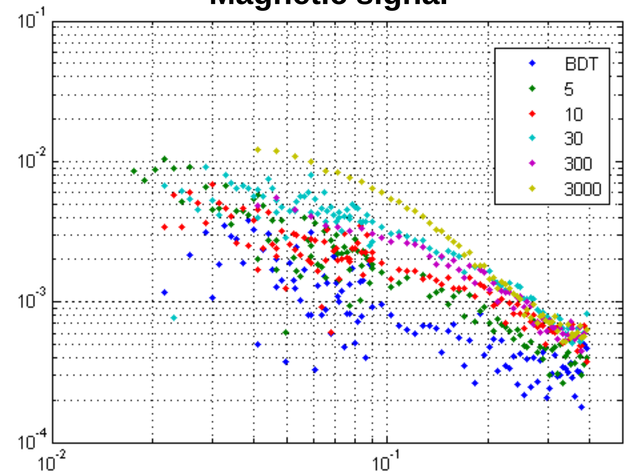
[DESCHAMPS A., DANOIX F., EPICIER T., DE GEUSER, F., PEREZ M. *PTM-Avignon*, F, (2010)]

Nb	C	N	S	O
0.080	< 0.0010 (4-4-3 ppm)	0.0189 (σ = 0.005)	<0.0010 (2-2-2 ppm)	< 0.0010 (6-4-4-7-ppm)

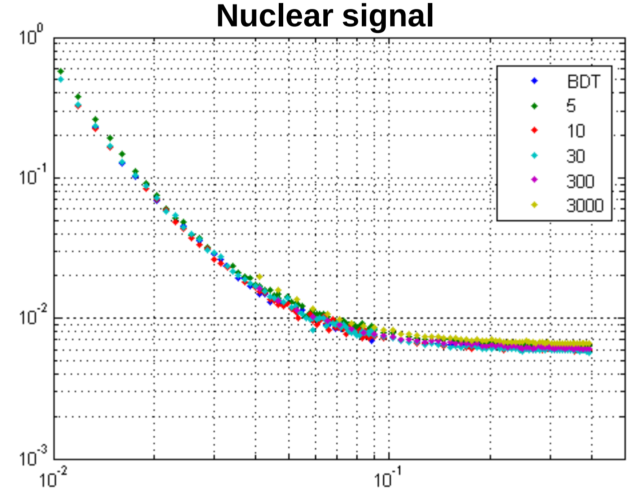
I.L.L.,
Grenoble-F



Magnetic signal

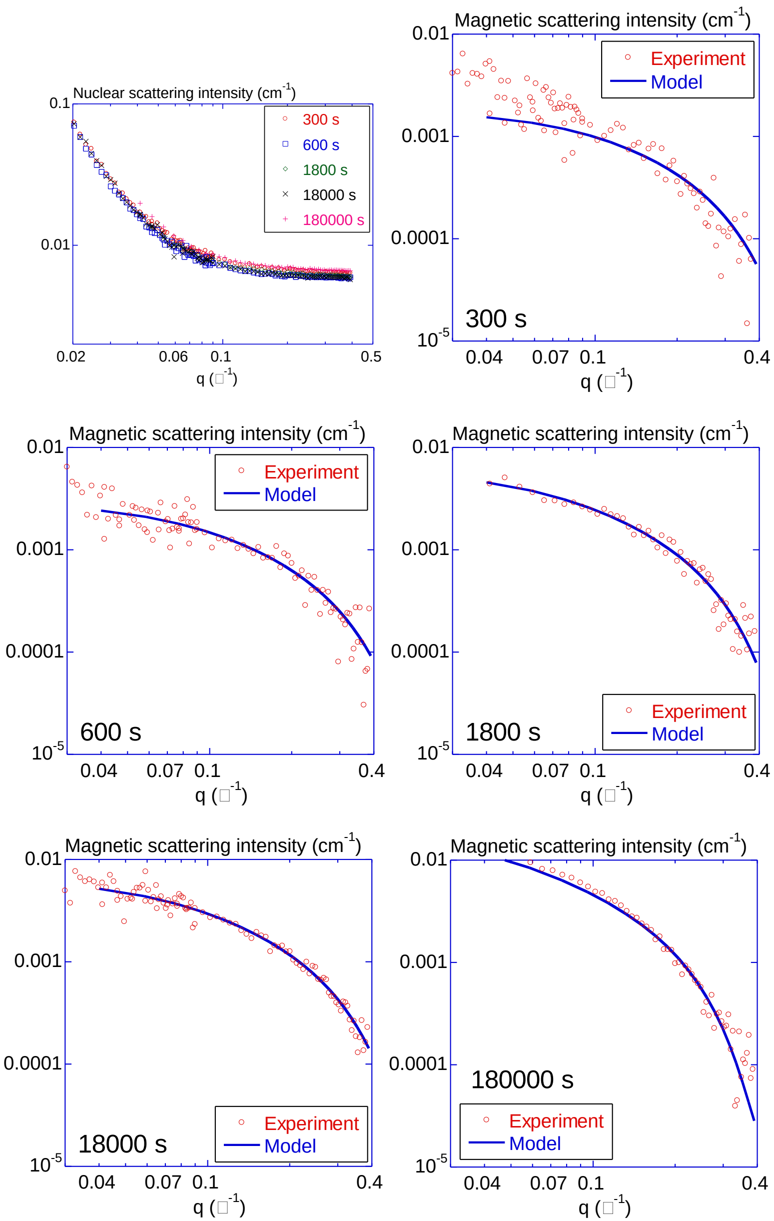


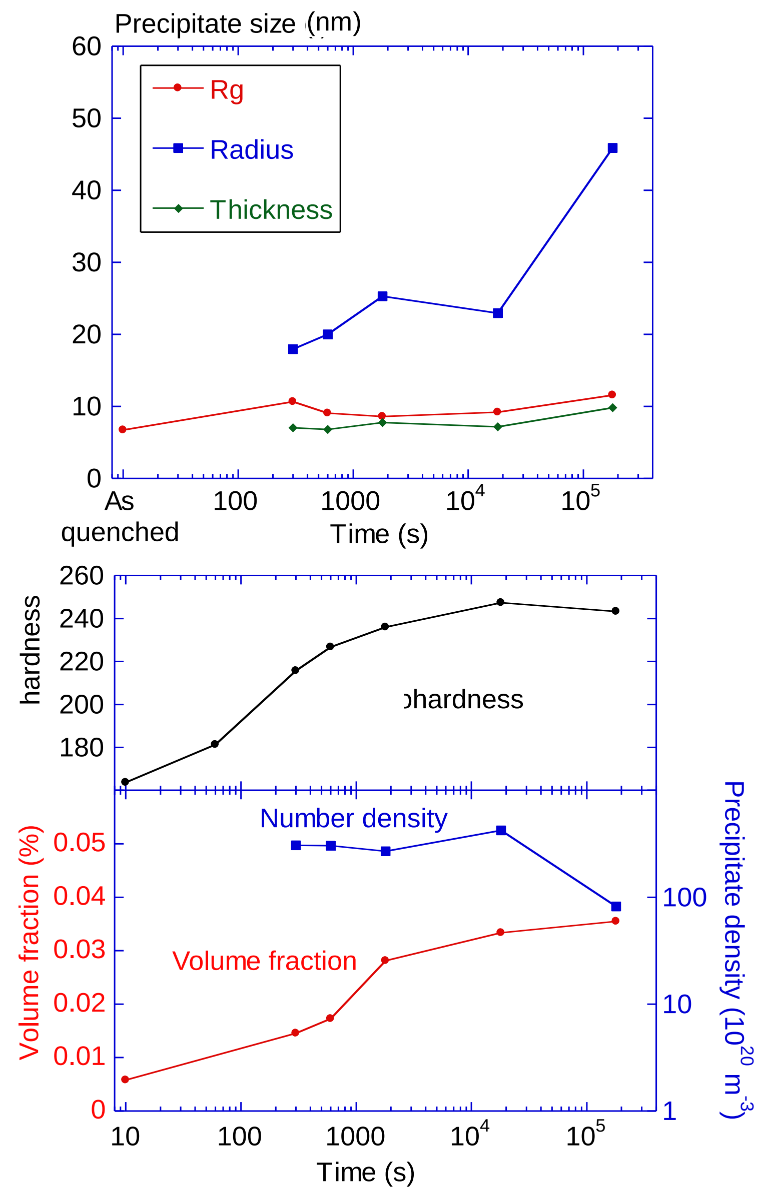
Nuclear signal



ANR Project O6|BLAN|0205

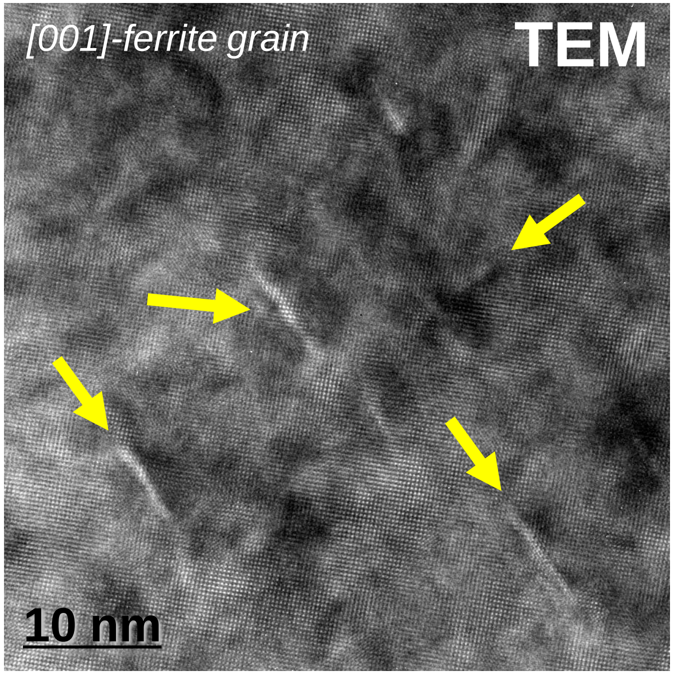
Guinier-Preston type ZONES in a pure Fe-Nb-N system



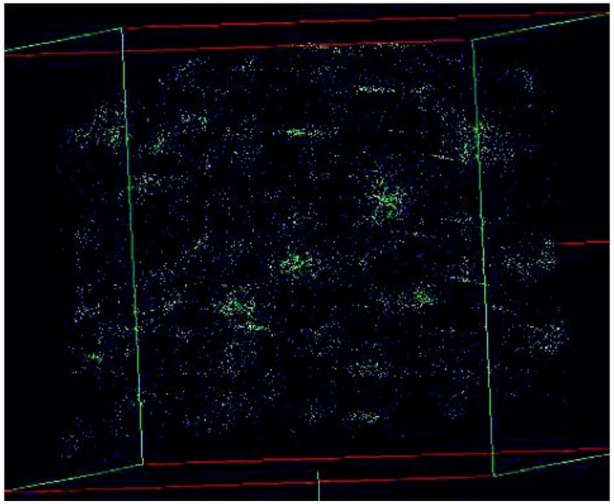


Guinier-Preston type ZONES in a pure Fe-Nb-N system

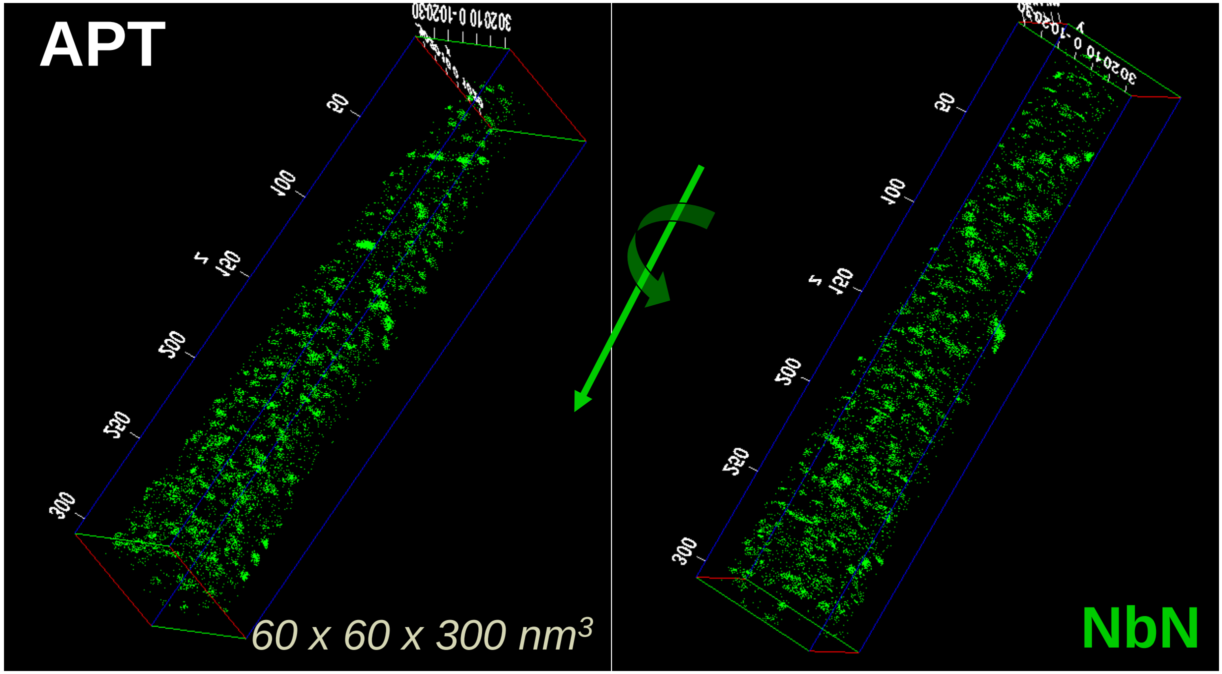
50 hrs @ 600° C



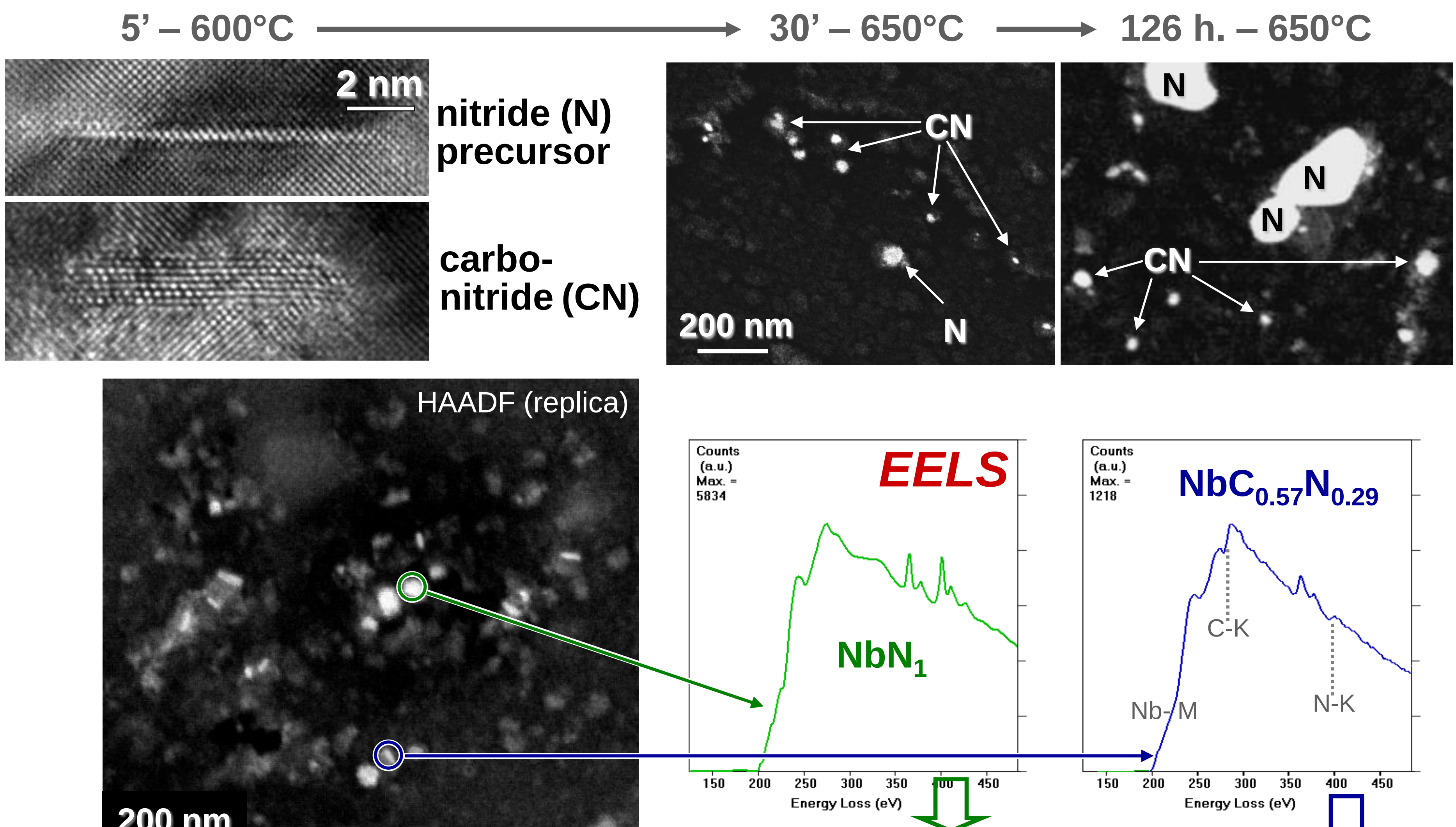
diameter close to ≈10 nm



80 x 80 x 70 nm^3



back to ***FeNbCN system…***



Replica (30' – 650°C)

pure NITRIDES Nb_1N_1

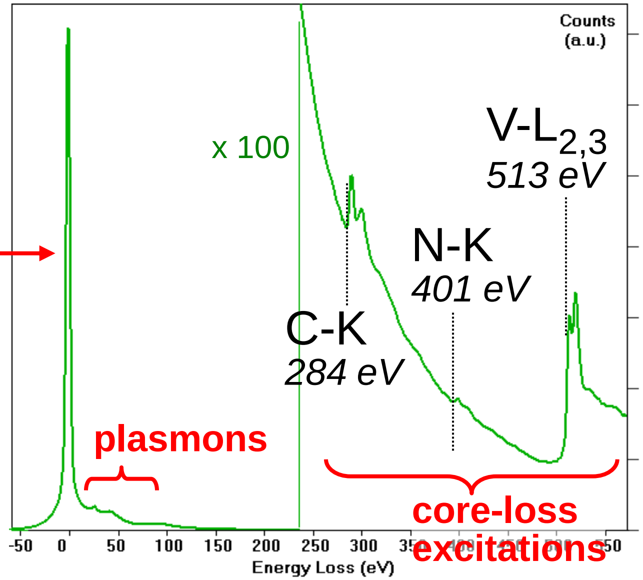
substoichiometric CARBO-NITRIDES

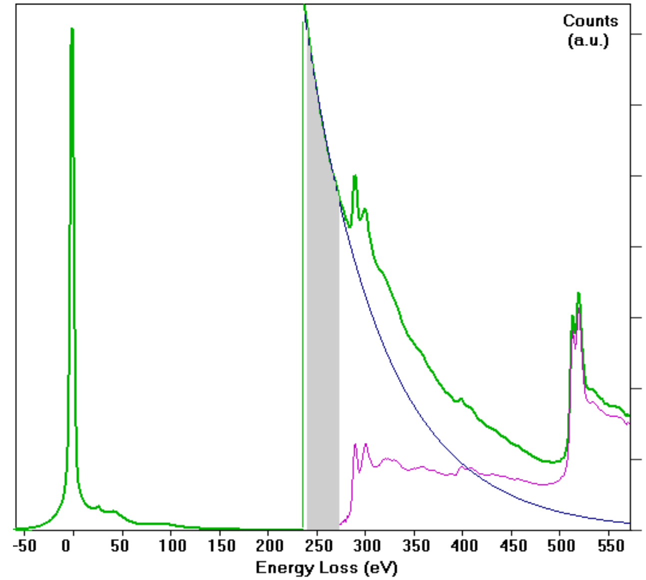
<u>average chemistry</u> $NbC_{0.58}N_{0.27}$

Introduction to EELS

[R.F. EGERTON, 'Electron Energy-Loss Spectroscopy in the Electron Microscope', 2nd Ed., Plenum Press (1996), 485 p.]

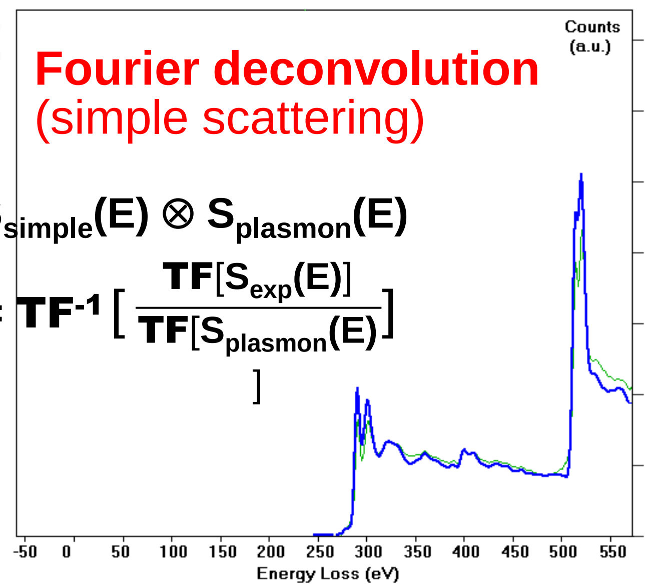
- **quantitative element analysis**





1 **background subtraction** power-law E^{-r}

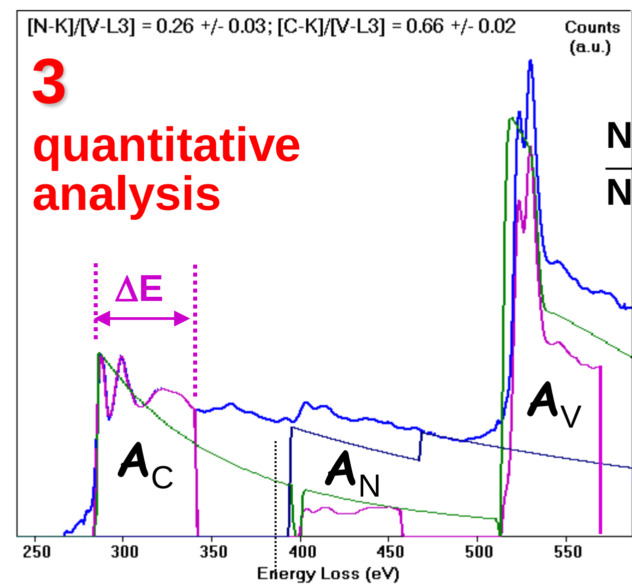
2 **Fourier deconvolution** (simple scattering)



$$S_{exp}(E) = S_{simple}(E) \otimes S_{plasmon}(E)$$

$$S_{simple}(E) = TF^{-1}\left[\frac{TF[S_{exp}(E)]}{TF[S_{plasmon}(E)]}\right]$$

3 **quantitative analysis**



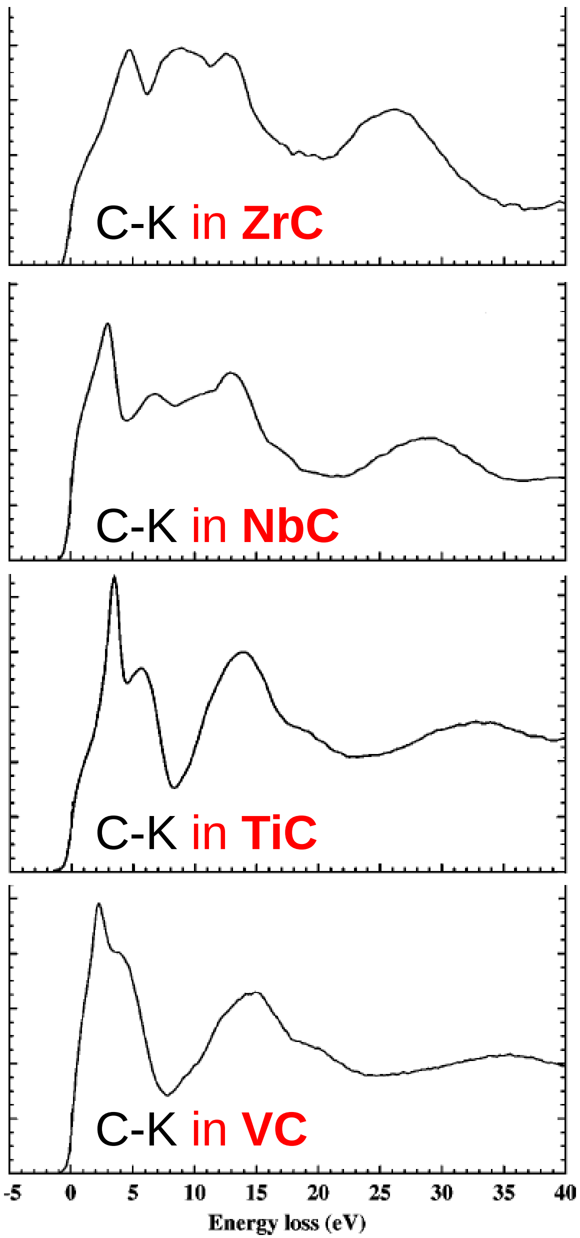
$$\frac{N_X}{N_Y} = \frac{A_X}{A_Y}\,\frac{\sigma_Y(\Delta E, \beta_{eff})}{\sigma_X(\Delta E, \beta_{eff})}$$

collection angle

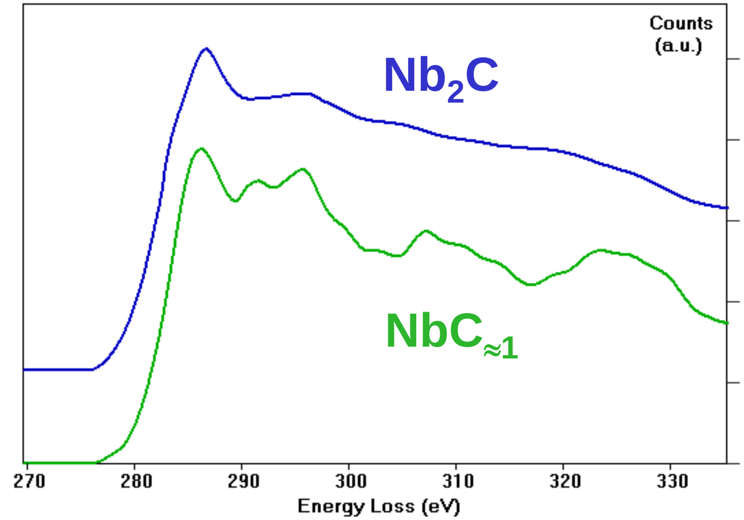
cross-section for inelastic scattering

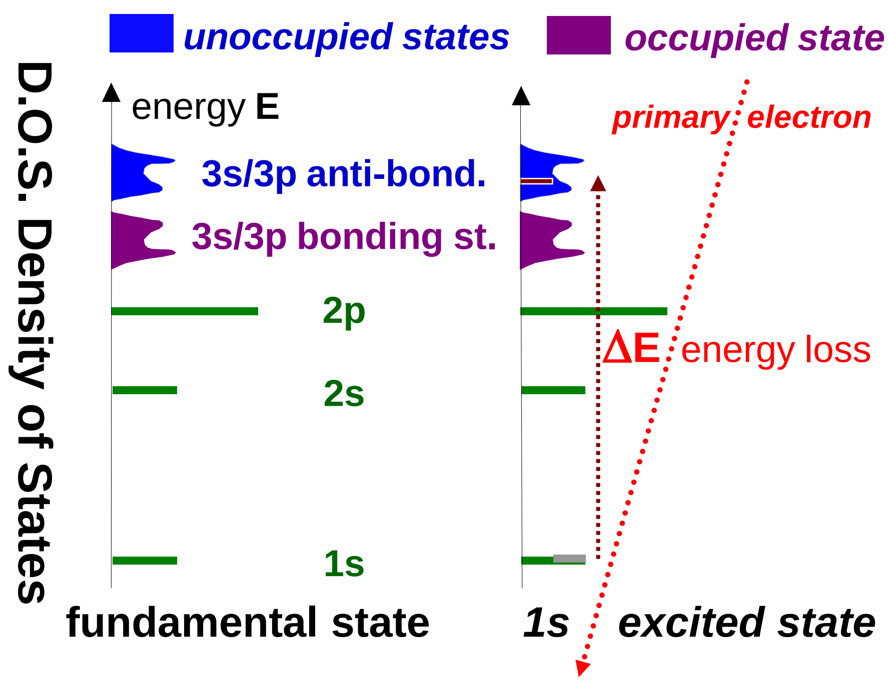
• Electron Energy-Loss Near-Edge fine Structures *(ELNES)*

[A.J. SCOTT et al., *Phys. Rev. B*, **63**, 245105, (2001)]

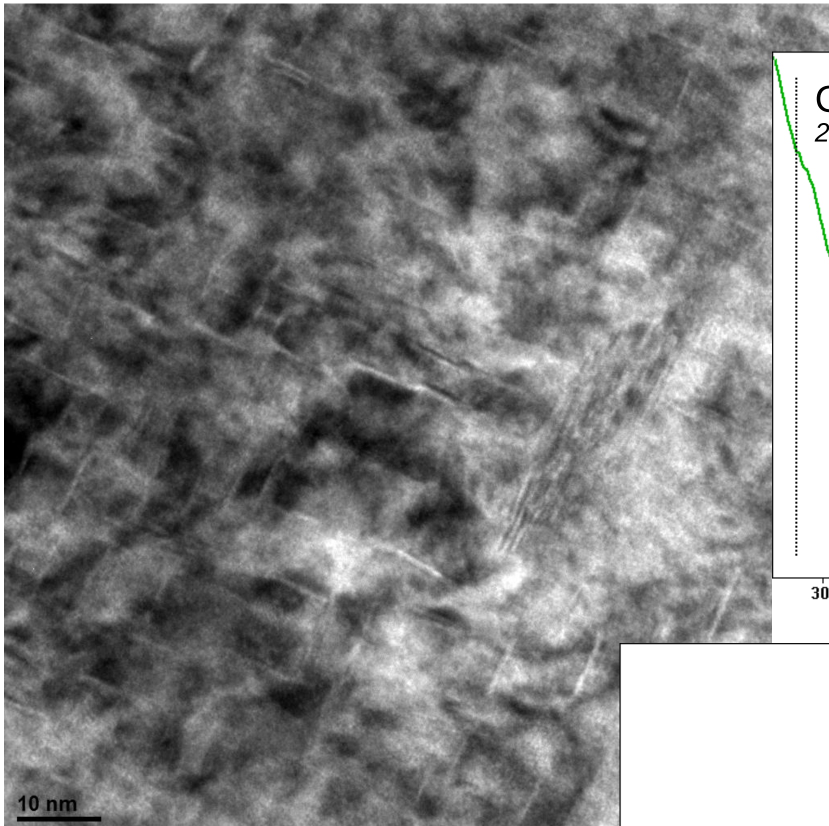


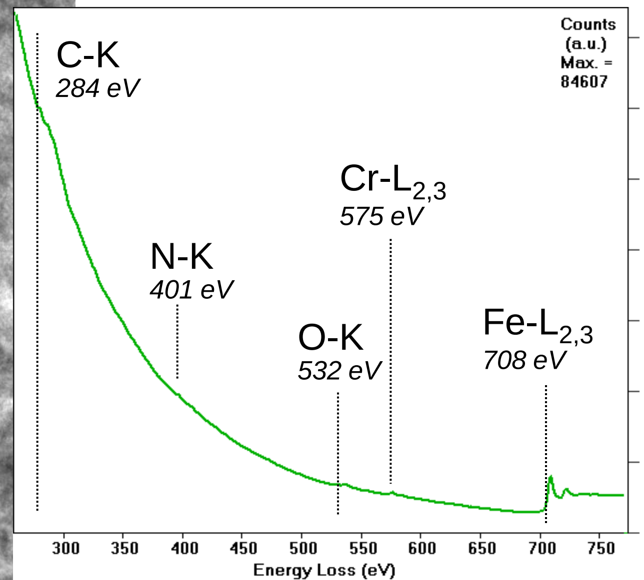
[T. EPICIER, E. COURTOIS, C. SCOTT, *unpublished*]

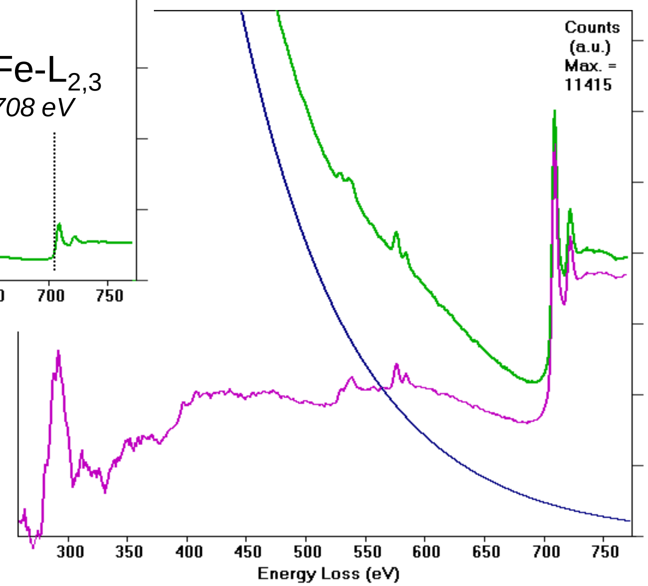


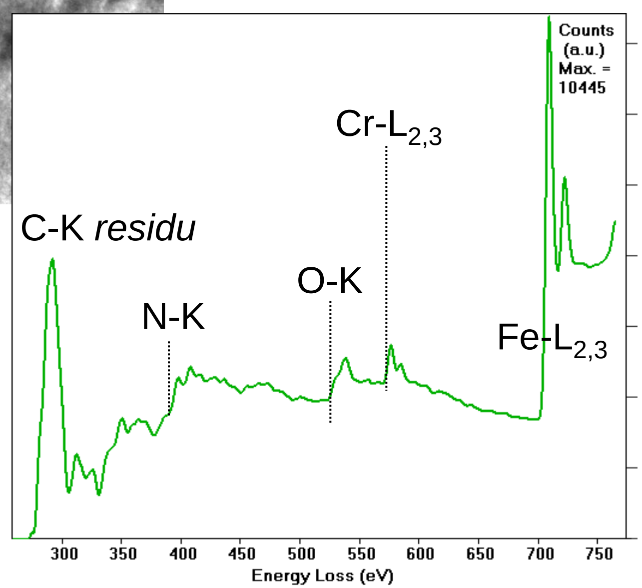


EELS in thin foils









FeCrN system

[P. JESSNER, PhD, GPM-Rouen]

inadequate background

- *too thick*
- *slight C contamination*
- ***cannot extract edge areas properly***

• EELS references

Nb-M from ***NbC$_{0.95}$, Nb$_6$C$_5$, NbN*** *(powders / bulk single- or poly-crystals)*

C-K *in NbC* from ***NbC$_{0.95}$, Nb$_6$C$_5$*** *(powders / single-crystal)*

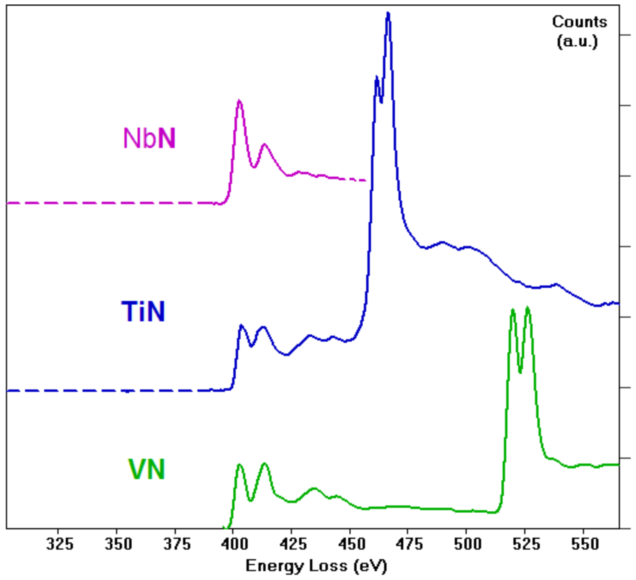
C-K *in VC* from ***V$_6$C$_5$*** *(bulk poly-crystals)*

C-K *in amorphous carbon* from C-film *(TEM grid)*

N-K *in NbN* from ***NbN*** *(powder)*

N-K *in TiN ≈ VN* from ***TiN*** *(precipitates)* and ***VN*** *(literature)*

Ti-L from ***TiN*** *(precipitates)*



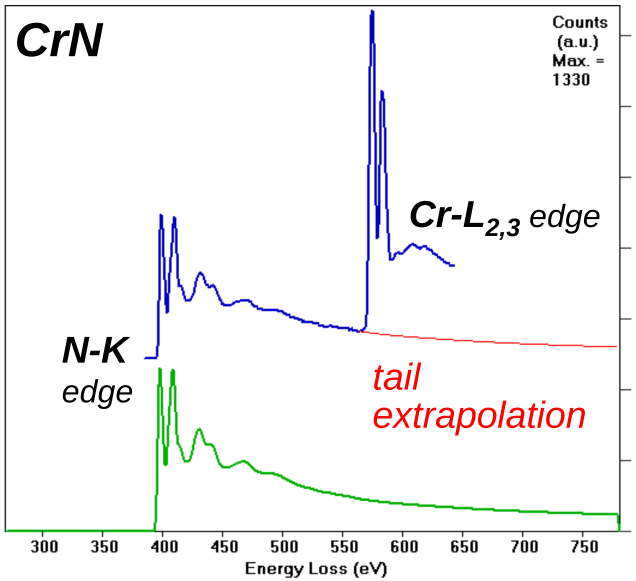
O-K *in Fe$_2$O$_3$* from ***oxidized Fe thin films***

Fe-L *in Fe$_2$O$_3$* from ***oxidized Fe thin films***

Cr-L from ***CrN, Cr$_2$N***

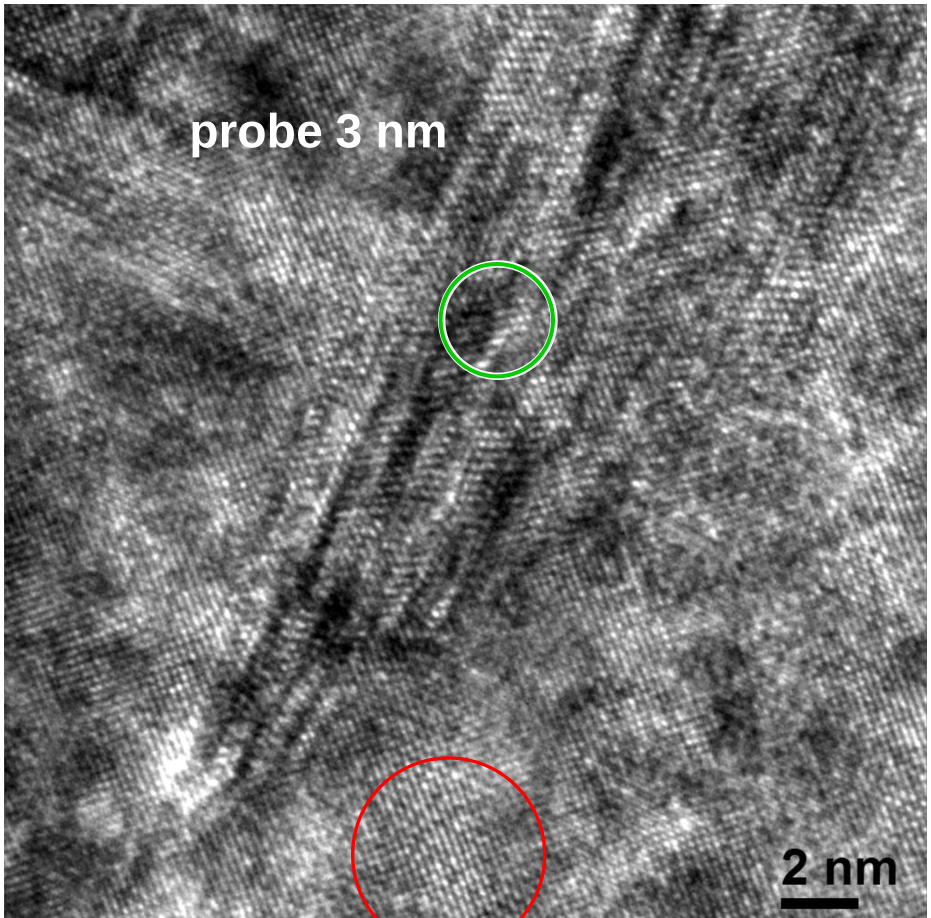
N-K *in CrN* from ***CrN***

[C. MITTERBAUER et al., *Sol. State Comm.*,**130**, (2004), 209–213]

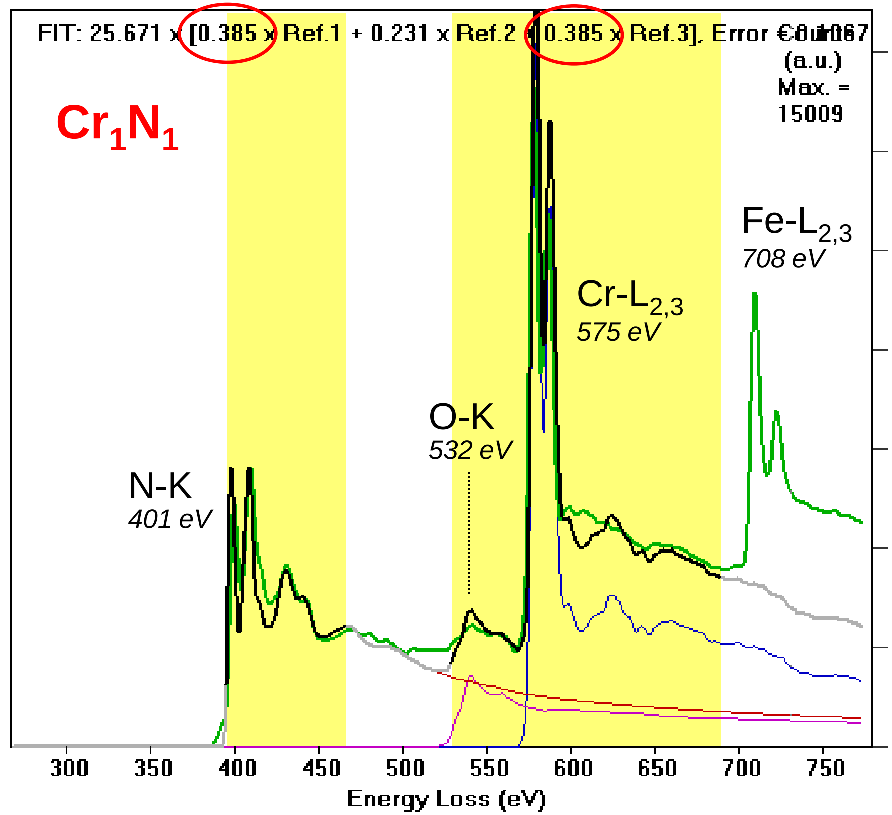


Quantitative **L**east-**M**ean **S**quare **F**itting
from normalized references

$Cr_xFe_?N_y$ *platelet*



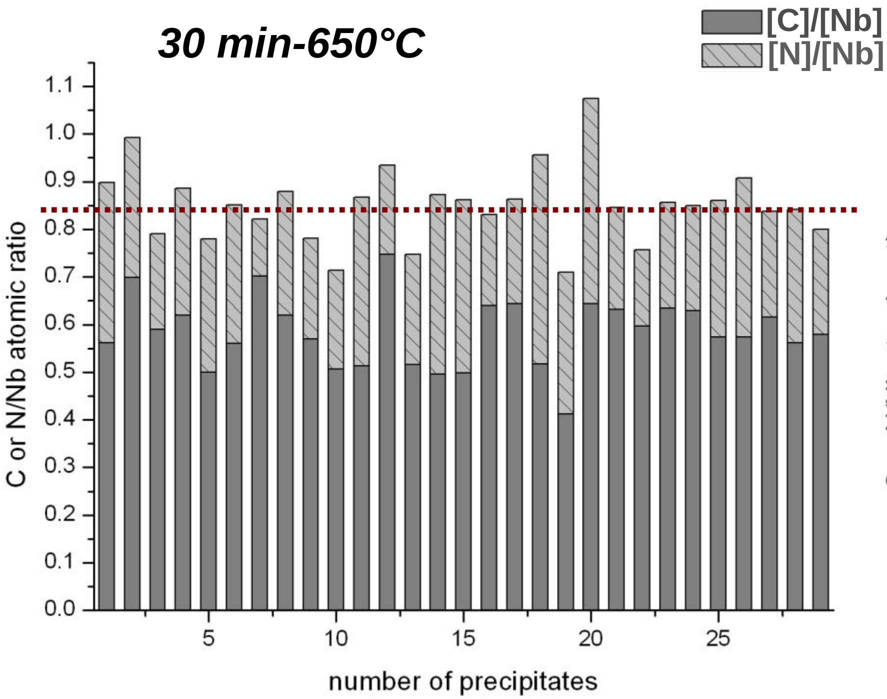
spurious oxidation



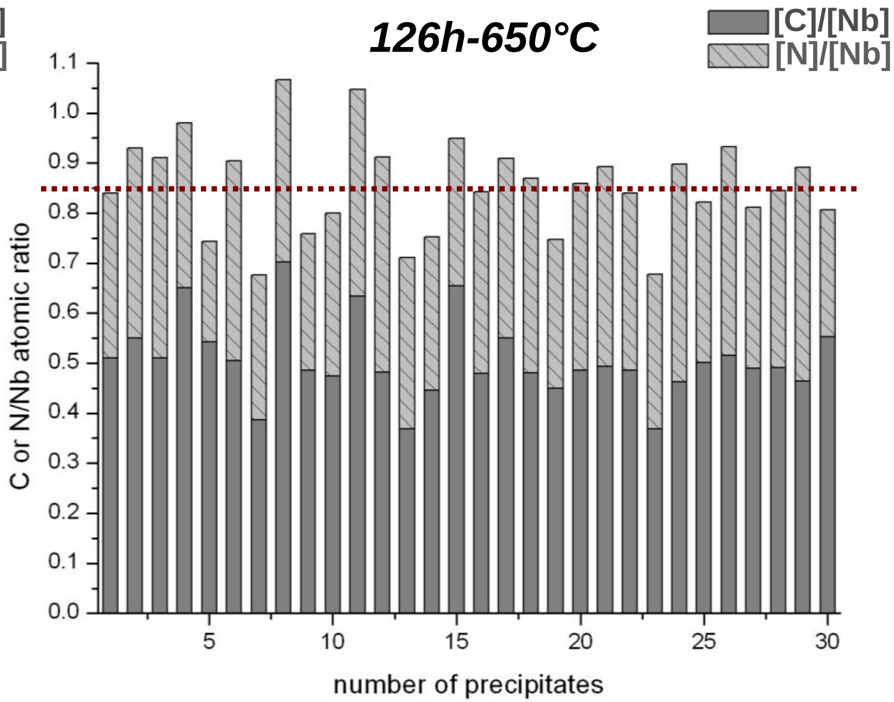
Quantitative **L**east-**M**ean **S**quare **F**itting from normalized references

◆ **Quantitative composition results for the CARBO-NITRIDE precipitates**

$Nb_1C_{0.59}N_{0.23}$ $\frac{[C+N]}{[Nb]} = 0.82$



$Nb_1C_{0.58}N_{0.27}$ $\frac{[C+N]}{[Nb]} = 0.85$



Application to complex mixed carbo-nitrides

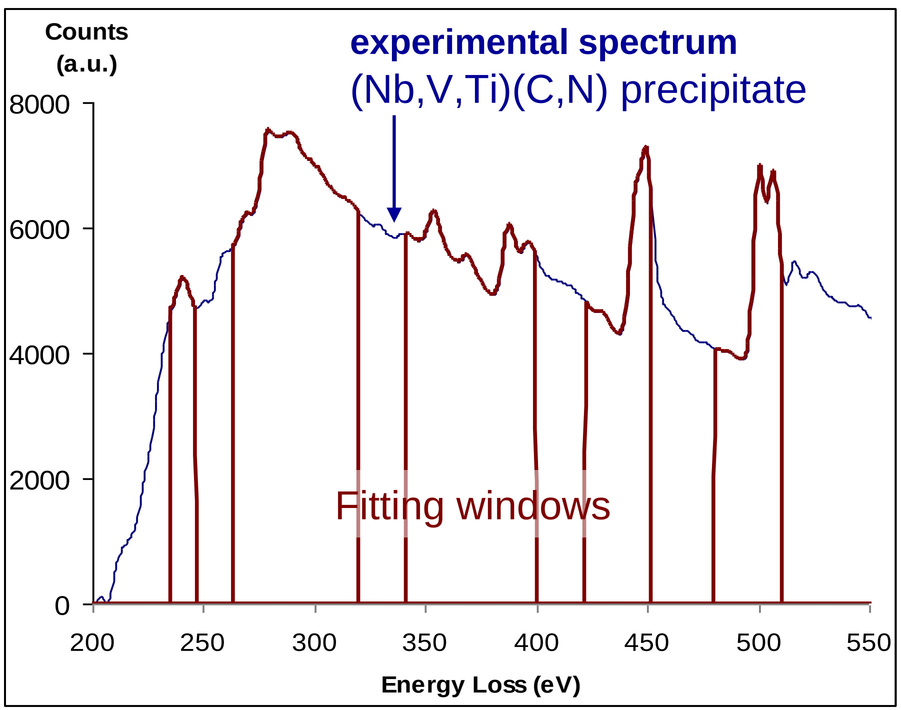
$$\text{precipitate} = \alpha Nb(C_{\alpha_1}N_{\alpha_2}) + \beta V(C_{\beta_1}N_{\beta_2}) + \gamma Ti(C_{\gamma_1}N_{\gamma_2}) + \varepsilon C_{am}$$

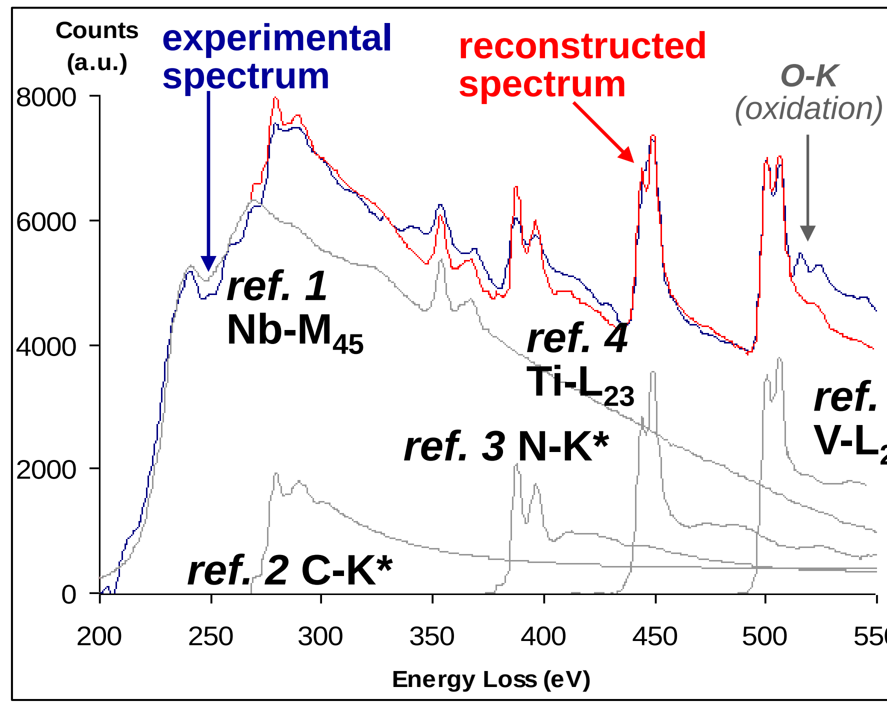
$$\alpha + \beta + \gamma = 1$$

$\varepsilon = 0$ if NO CONTAMINATION

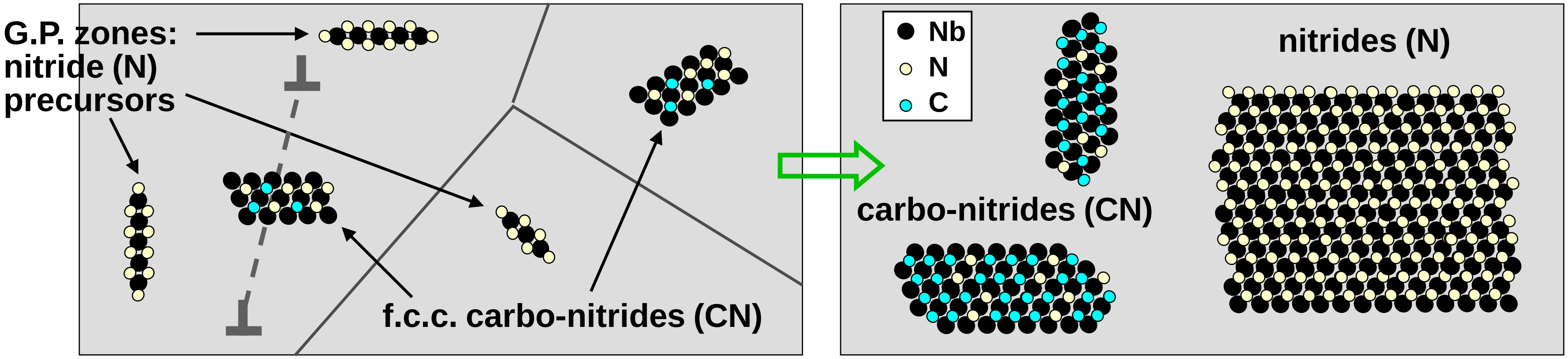
$$0.5 \le \alpha_1 + \alpha_2 \le 1$$
$$0.5 \le \beta_1 + \beta_2 \le 1$$
$$0.5 \le \gamma_1 + \gamma_2 \le 1$$

$y \in [\approx 0.5, 1]$ in cubic MX_y carbonitrides



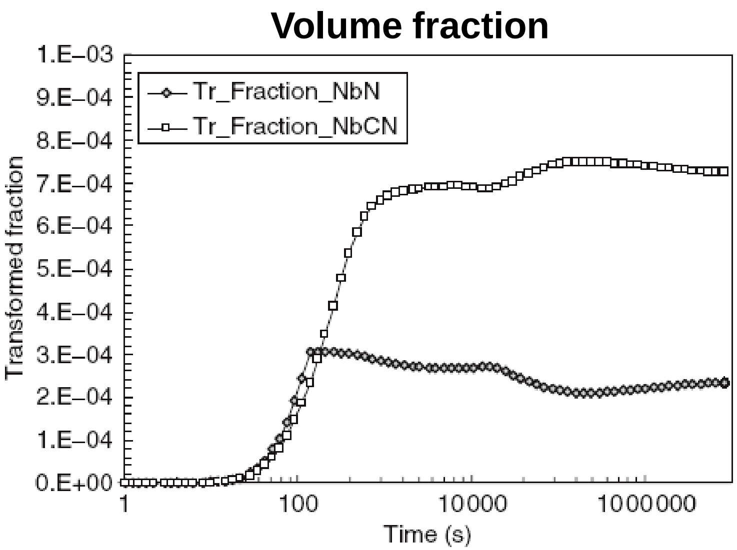


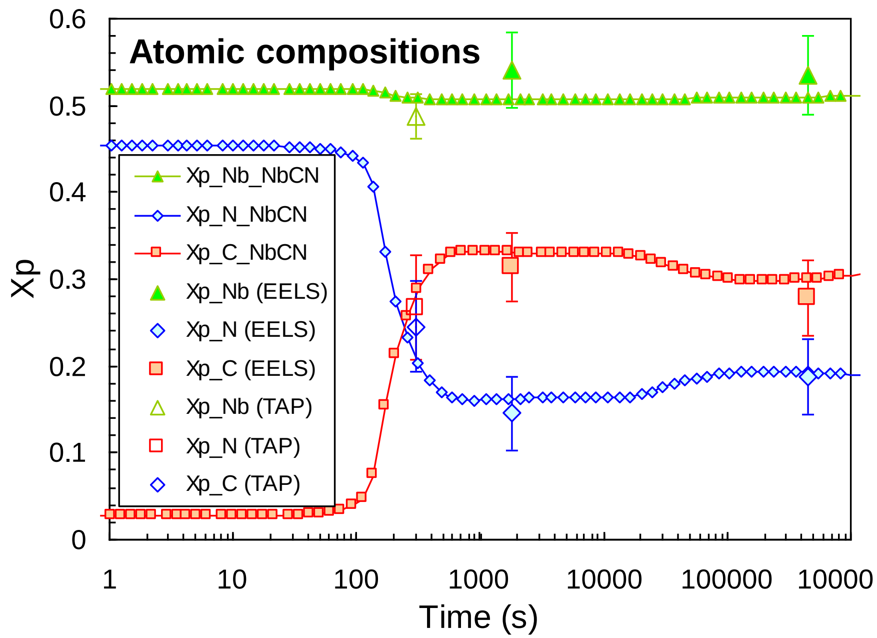
[LEGUEN C, *PhD thesis*, INSA-Lyon, (2010)]



◆ MODELLING of NbN + NbCN populations

[M. PEREZ, É. COURTOIS, D. ACEVEDO, T. ÉPICIER, P. MAUGIS, *Phil. Mag. Letters* **87**, 9, 645, (2007)]





Precipitation in the FeNbVC system

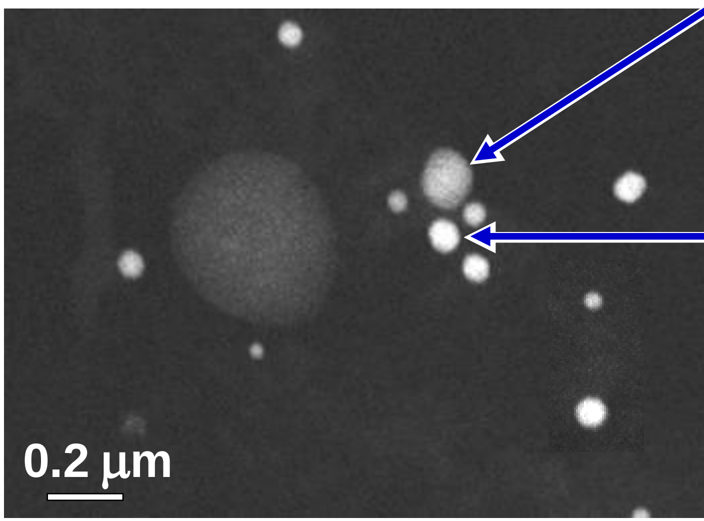
ASCOMETAL

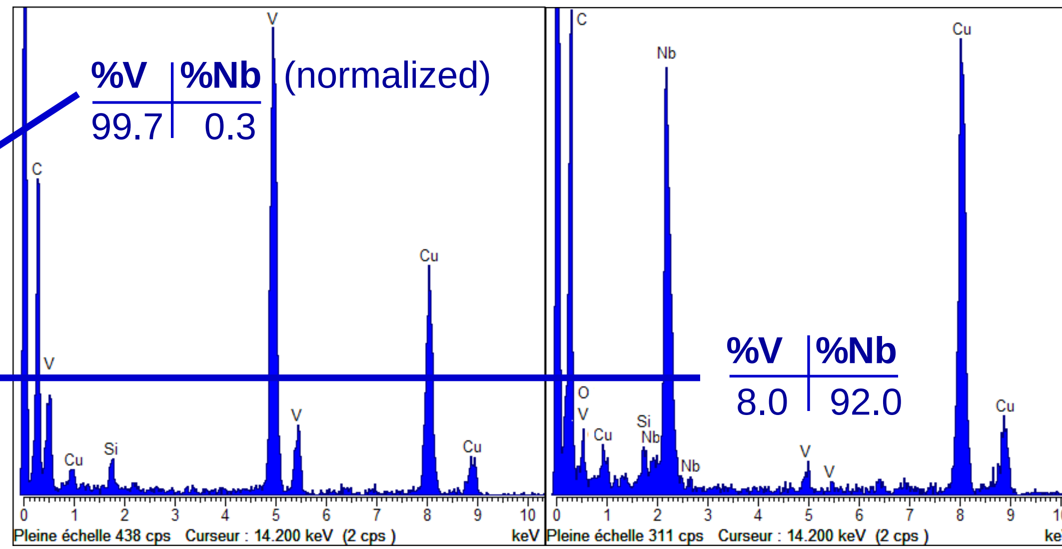
[D. ACEVEDO, *PhD thesis*, INSA Lyon, (2007)]
[D. ACEVEDO, M. PEREZ, T. EPICIER et al., *Min., Metals & Mater. Soc.*, (2009)]

◆ A SUMMARY…

- TEM analysis (EDX, **HAADF**) demonstrates that the dissolution of precipitates involves the co-existence of ***TWO populations: V-rich and Nb-rich carbides***

STEM-HAADF (6 days @ 950°)

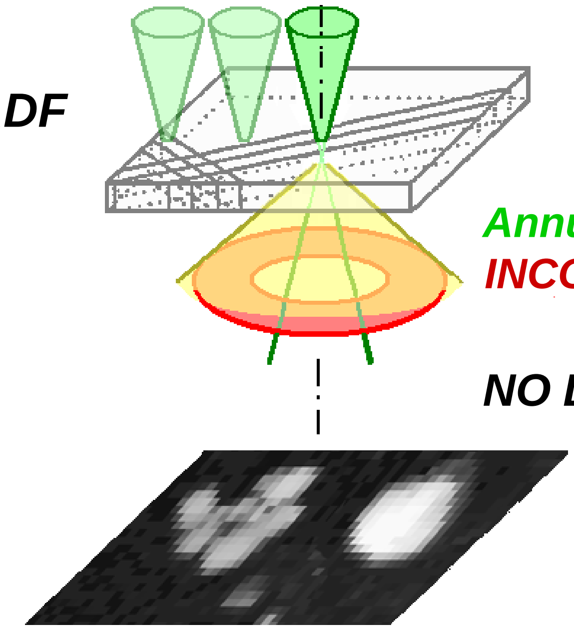




- **Thermodynamical modelling** confirms this process

• STEM High Angle Annular Dark Field *for CHEMISTRY*

- Background on HAADF



Annular detector → collection of INCOHERENT electron scattered at high angle

NO DYNAMICAL SCATTERING

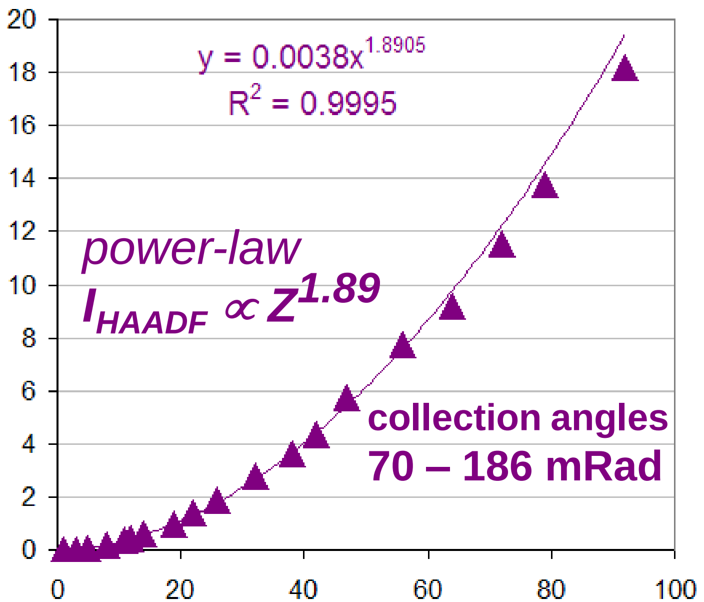
- Experimental conditions

$$\mathbf{f_{atom}(q) = \frac{1}{2\pi^2 a_0 q^2} Z}$$

(Rutherford scattering)

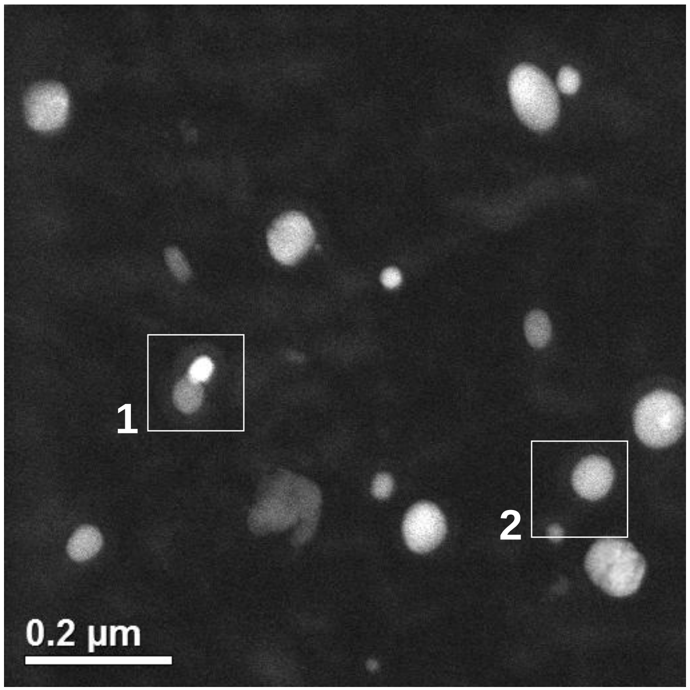
($q = \frac{2\sin(\theta)}{\lambda}$, $a_0 = \varepsilon_0 h^2/\pi m_0 e^2$ - *Bohr's radius* -, Z : atomic number)

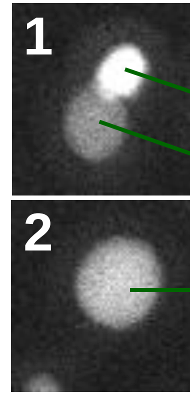
$f^2_{atom}(q) \propto Z^2$ and $I_{HAADF}(q)$ *roughly* $\propto Z^2$



• Quantitative chemistry in ‘mixed’ $(V_{1-x}Nb_x)C$ precipitates

extraction C-replica of **(V,Nb)C** precipitates within ferrite

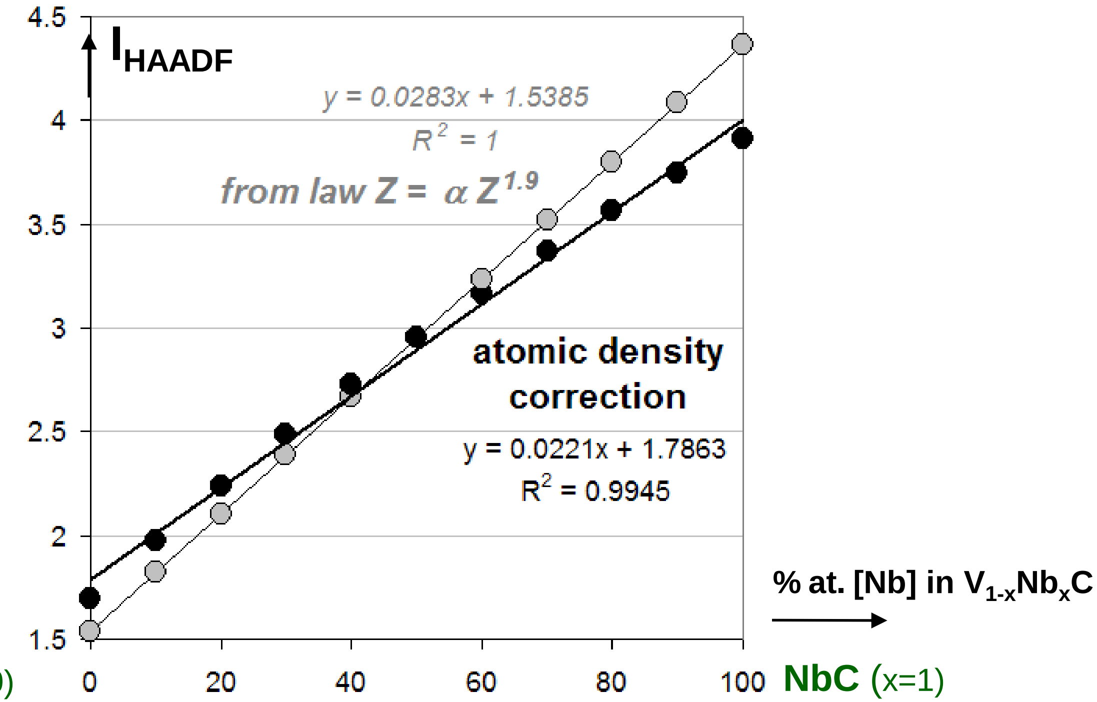


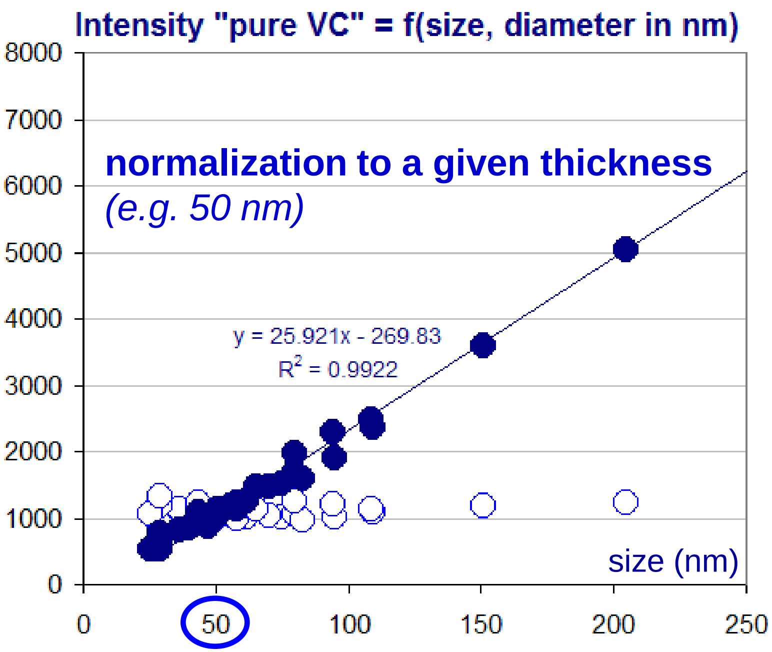


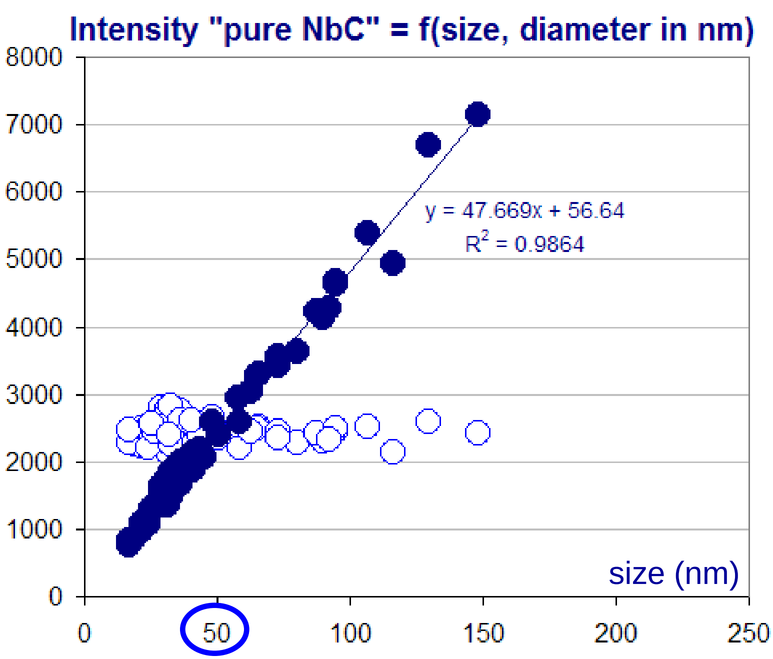
	%V	%Nb	(at. normalized)
1	75.1	24.9	*EDX analysis*
1	100	0	
2	97.7	2.3	

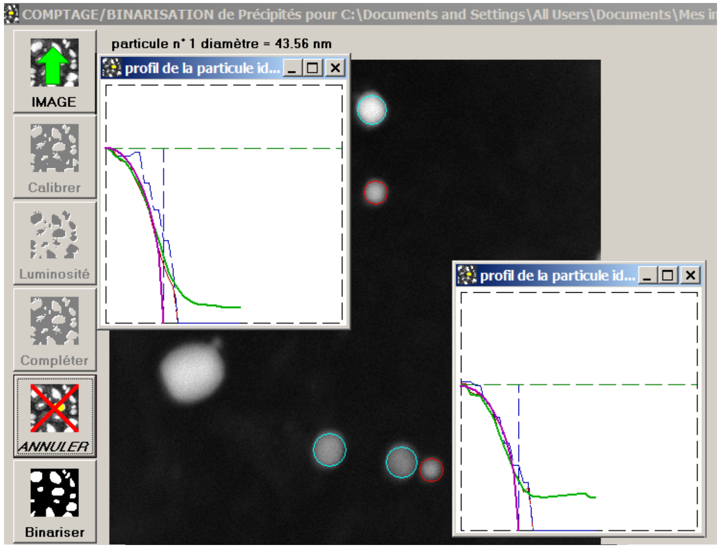
$I_{HAADF} \propto Z^{\alpha}$ (α = 1.7 – 2)

the HAADF intensity is linearly linked to the chemical composition for a *given* *mass-thickness*







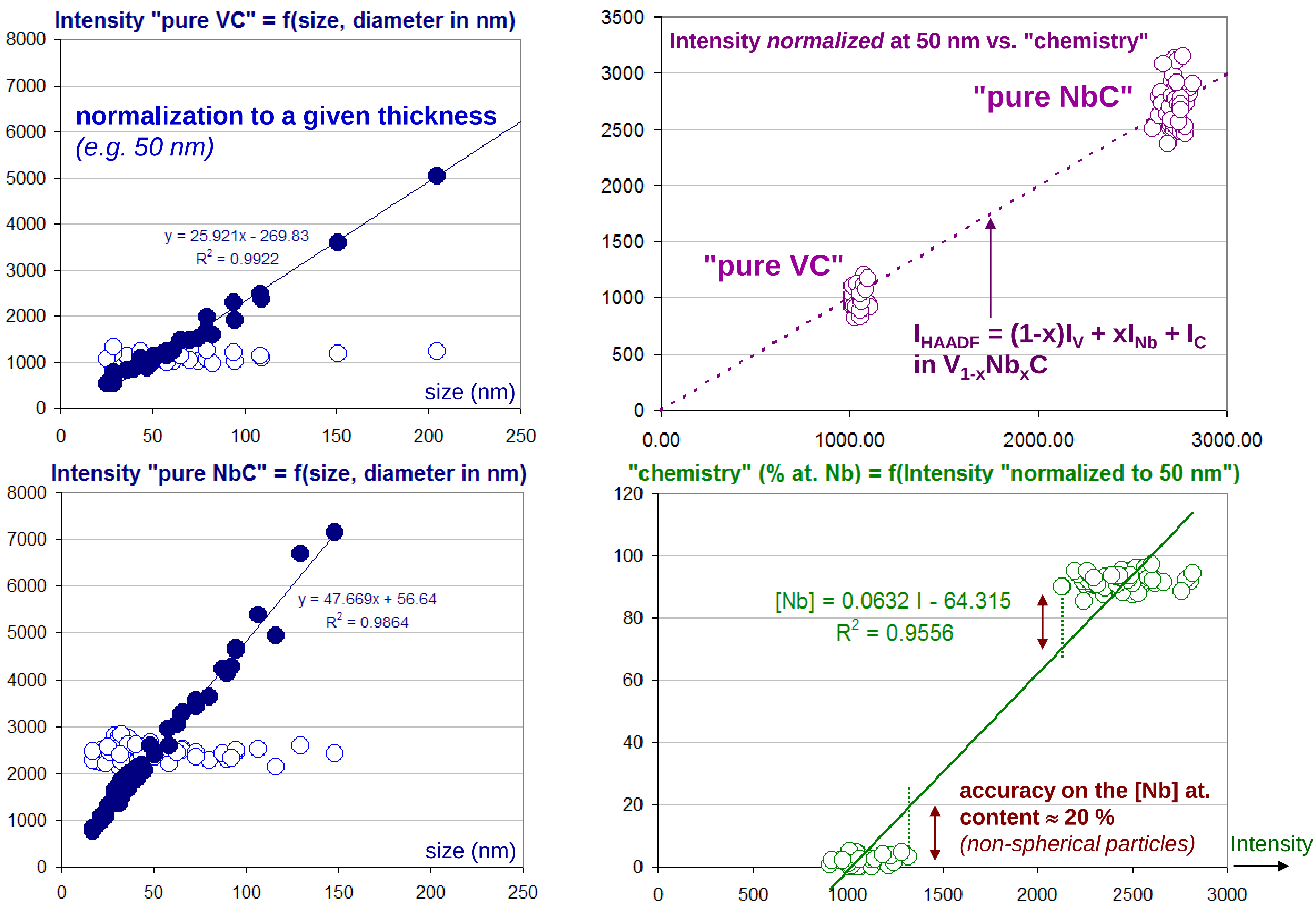


dedicated software to analyse the HAADF images

(quantitative measurement of the intensity assuming spherical particles)

précipité

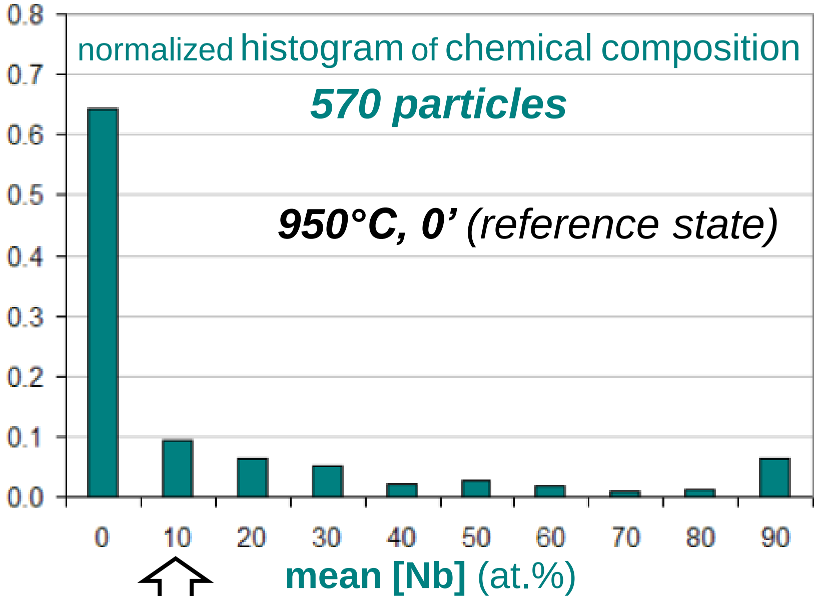
[EPICIER T., TOURNUS F., SATO K., KONNO T., *IMC17*, (2010)]

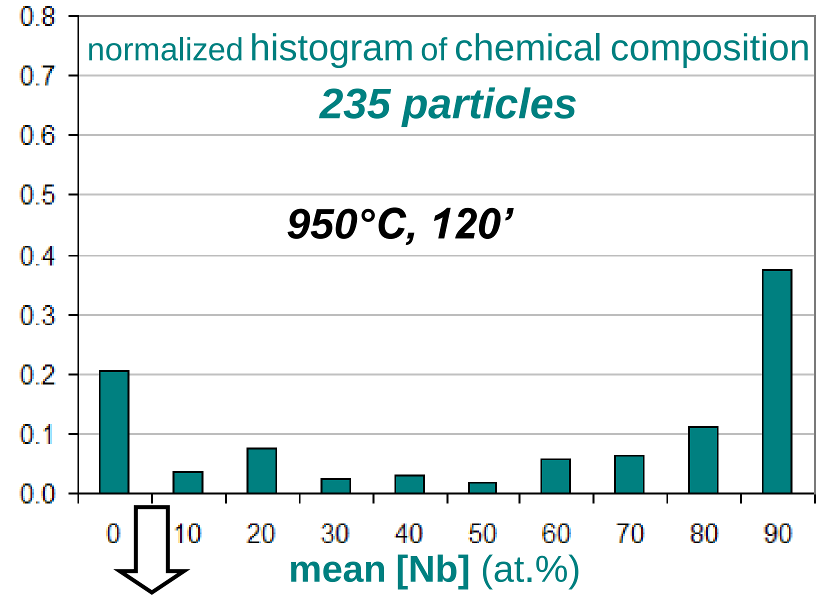


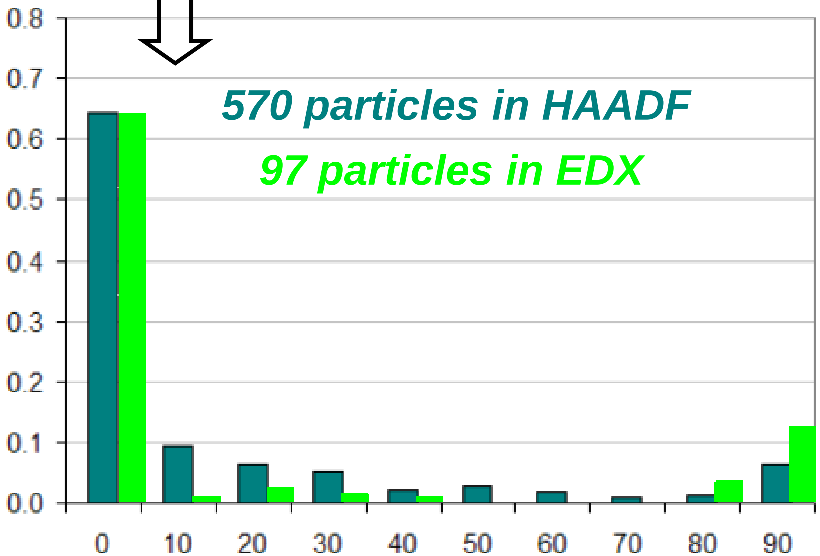
Intensity "pure VC" = f(size, diameter in nm)
normalization to a given thickness
(e.g. 50 nm)
y = 25.921x - 269.83
R2 = 0.9922
size (nm)
Intensity normalized at 50 nm vs. "chemistry"
"pure NbC"
"pure VC"
I_HAADF = (1-x)I_V + xI_Nb + I_C
in V_1-xNb_xC
Intensity "pure NbC" = f(size, diameter in nm)
y = 47.669x + 56.64
R2 = 0.9864
size (nm)
"chemistry" (% at. Nb) = f(Intensity "normalized to 50 nm")
[Nb] = 0.0632 I - 64.315
R2 = 0.9556
accuracy on the [Nb] at. content ≈ 20 %
(non-spherical particles)
Intensity

- ***Illustration (alloy Fe-(V,Nb)C: reversion at 950°C)***

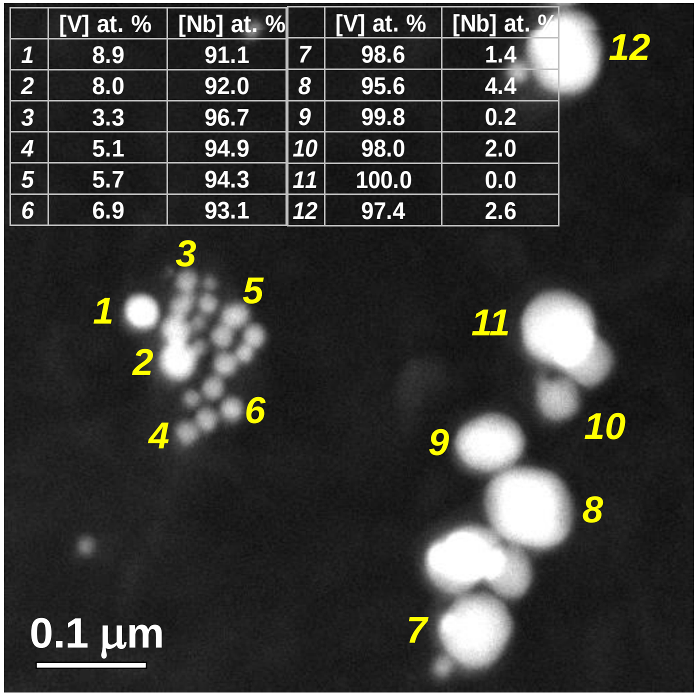
ASCOMETAL







detail EDX



	[V] at. %	[Nb] at. %		[V] at. %	[Nb] at. %
1	8.9	91.1	*7*	98.6	1.4
2	8.0	92.0	*8*	95.6	4.4
3	3.3	96.7	*9*	99.8	0.2
4	5.1	94.9	*10*	98.0	2.0
5	5.7	94.3	*11*	100.0	0.0
6	6.9	93.1	*12*	97.4	2.6

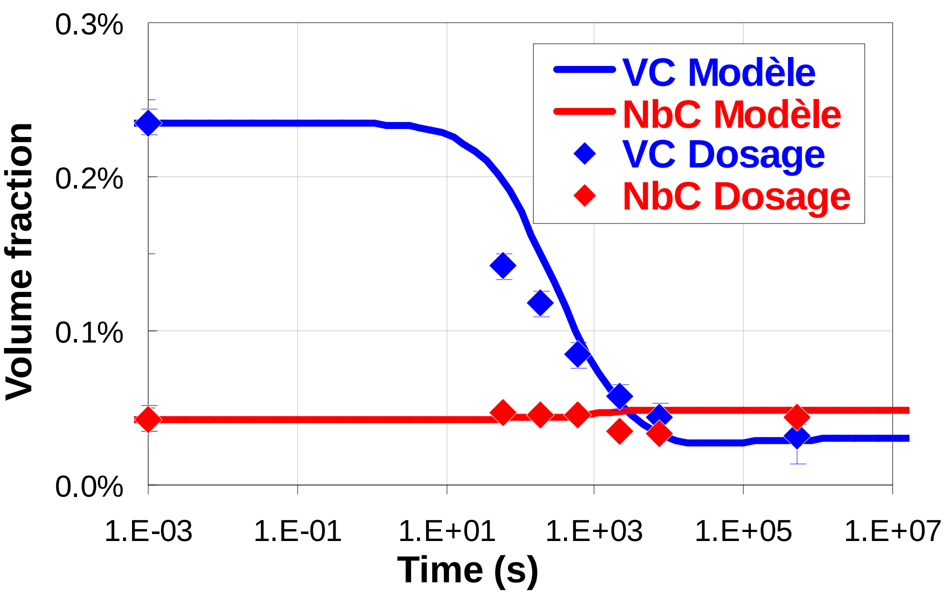
LARGE V-rich particles, SMALL Nb-rich particles

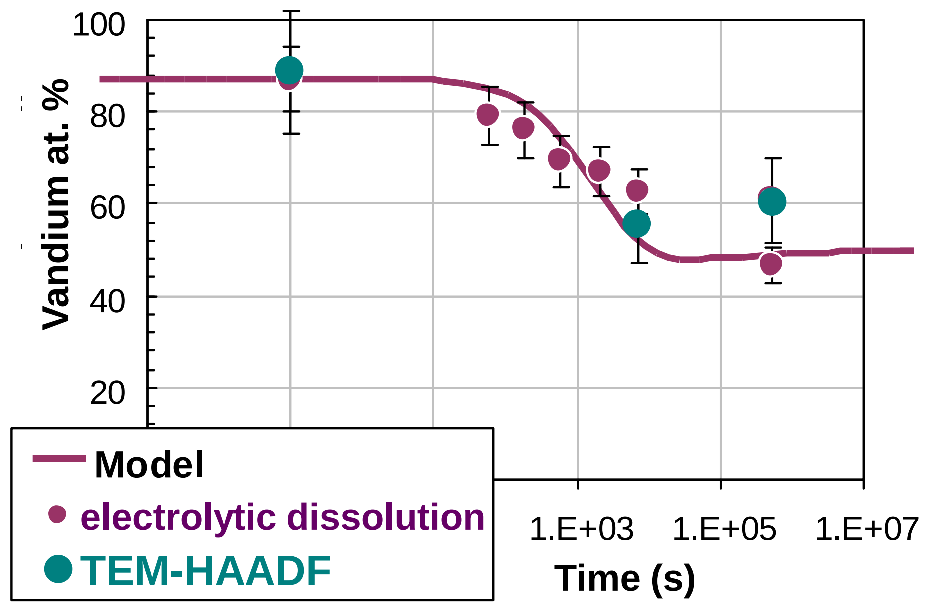
• ***Thermodynamical modelling***

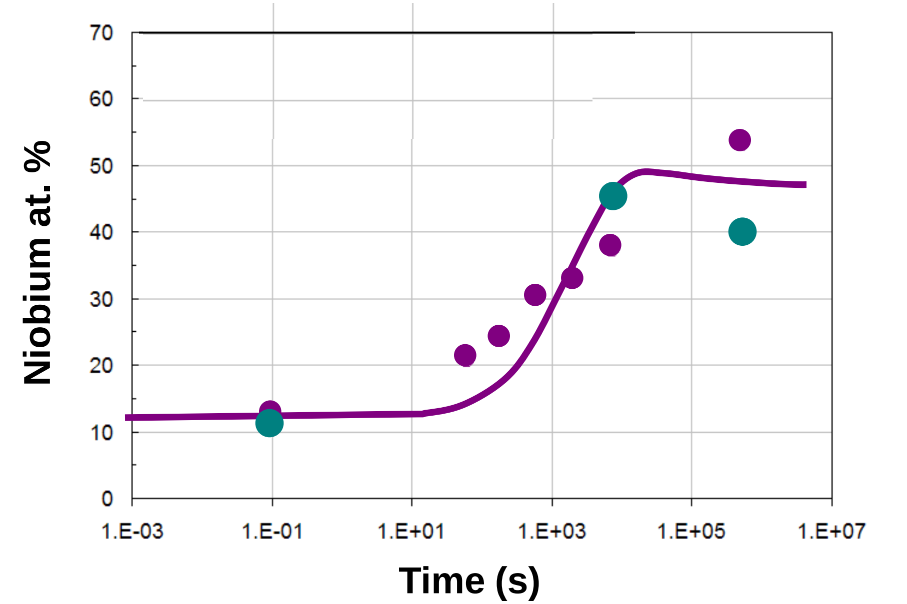
ASCOMETAL

global enrichment in [Nb] as a function of time:

coarsening / dissolution of VC-rich precipitates







Acnowledgements

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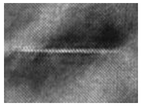
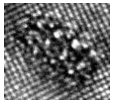
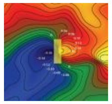
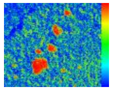
- Frédéric DANOIX, GPM-Rouen **(AP)FIM** 

- Églantine COURTOIS, Michel PEREZ, Claire LEGUEN, MATEIS Lyon **système FeNbCN** 

- Rachid EL BOUAYADI, Daniel ARAUJO, MATEIS Lyon **HAADF quantitatif** 

- Frédéric De GUEUSER, Williams LEFEBVRE, GPM Rouen **alliages Al-(Mg,Si)** 

- J. DOUIN, CEMES Toulouse, Patricia DONNADIEU, SIMAP Grenoble **images GPA** 

- Pascale BAYLE-GUILLEMAUD, CEA Grenoble, Béatrice VACHER, ECL Écully **EFTEM** 

- Gilbert THOLLET, Annie MALCHÈRE, Agnès BOGNER, Daniel ACEVEDO, MATEIS Lyon **MEB/ESEM**