

# Examples of application in metallurgy

## 1) Ferrite nucleation in deformed austenite

(M. Veron & S. Lacroix, 2003)

## 2) TRIP Steels

Multiphased TRIP steels, Nippon Steel, E Rauch @SIMAP

Stainless austenitic TRIP steels, M. Véron Coll. D. Embury, K. Spencer, P. Jacques



## 3) Recrystallization of ferritic stainless steels

Collaboration with N. Meyer, Ugitech

## 4) Thin Films

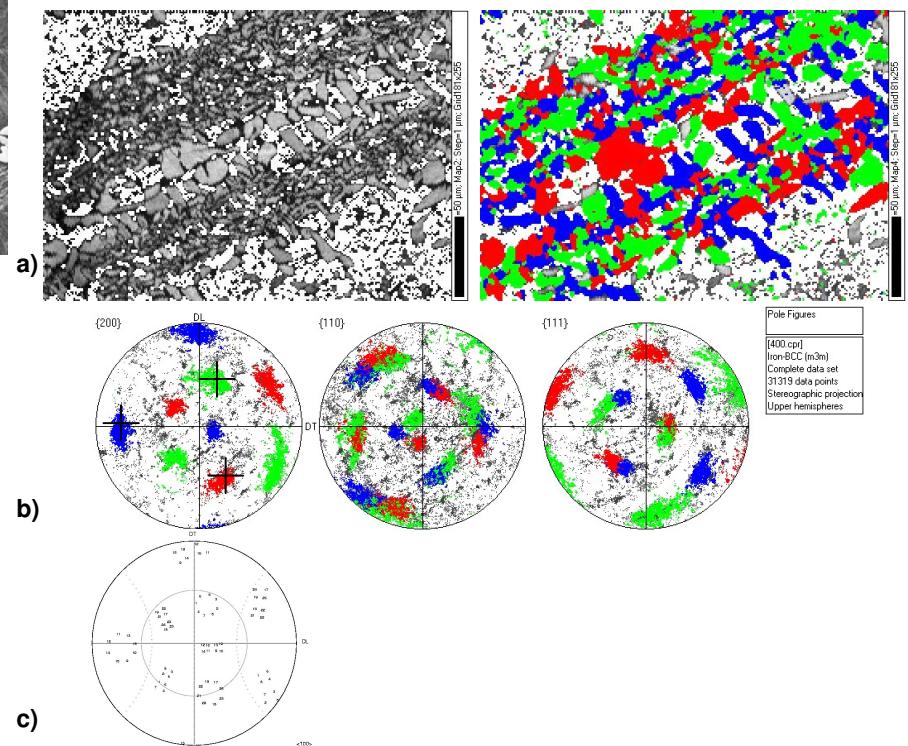
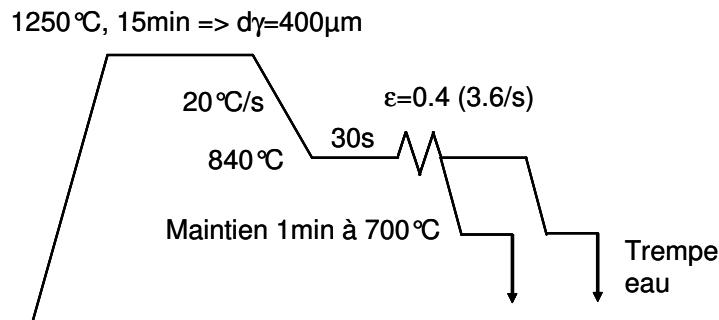
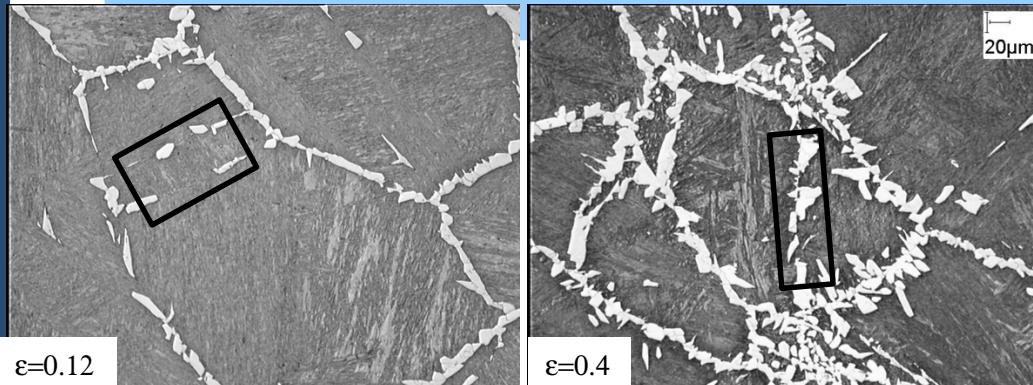
S. Lay, SIMAP. + Collaboration with S. Godet, ULB, Bruxelles

## 5) Sintered materials : WC-Co

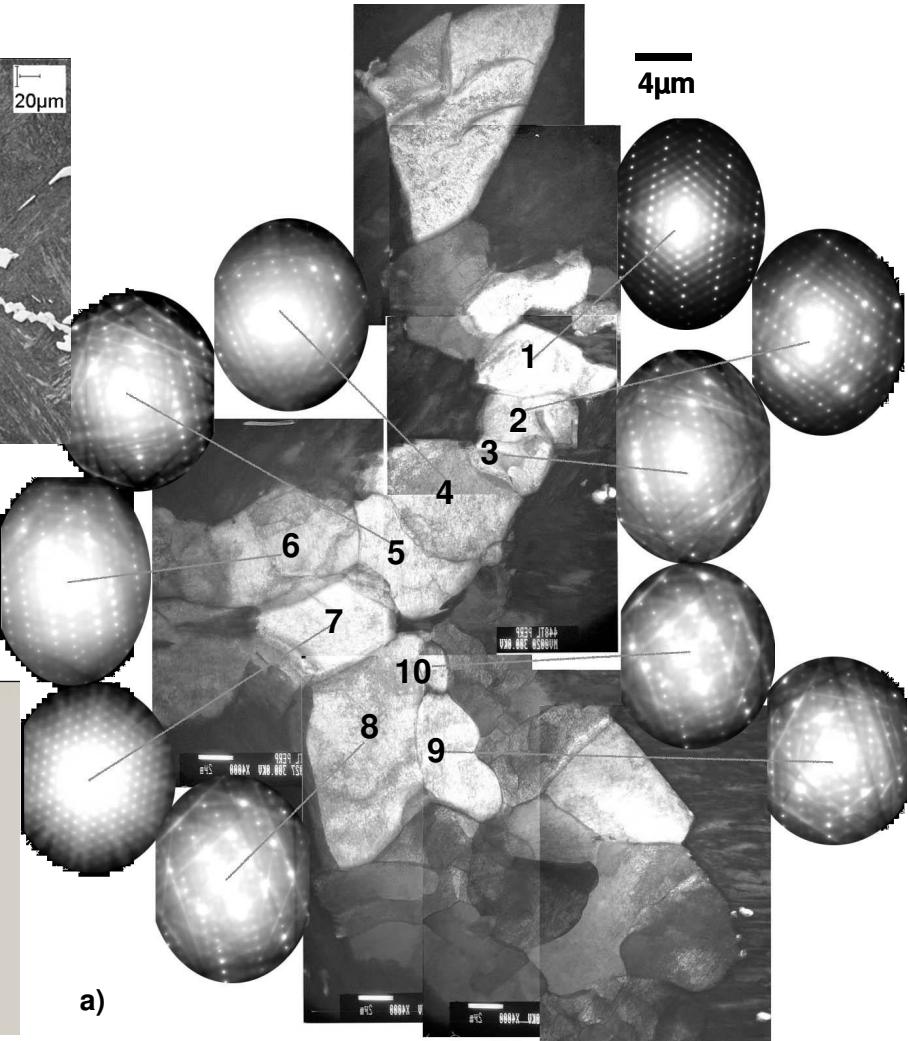
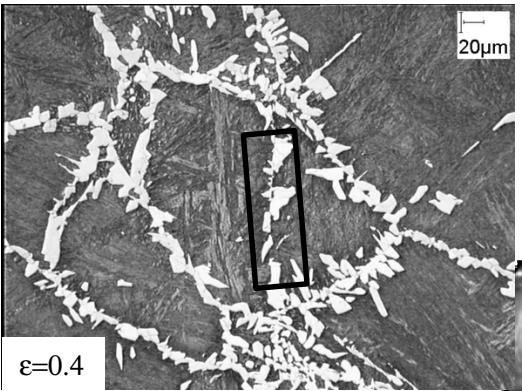
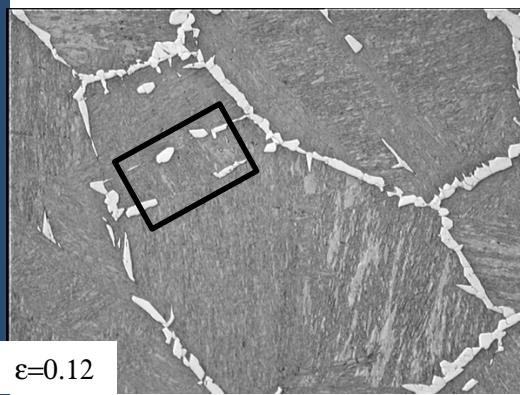
S. Lay, SIMAP. Pr JIN, Pekin, Mat science lab.

# 1) Ferrite nucleation in deformed austenite

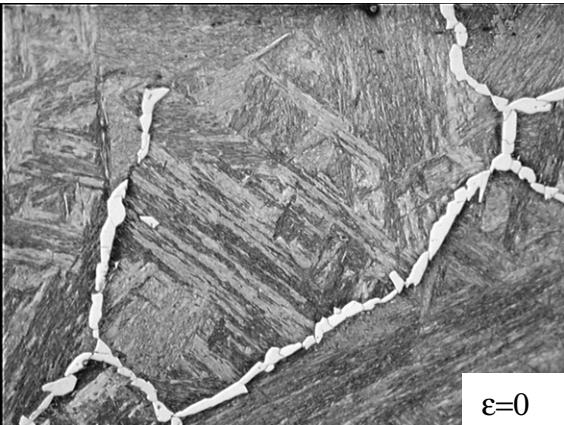
(M. Veron & S. Lacroix, 2003)



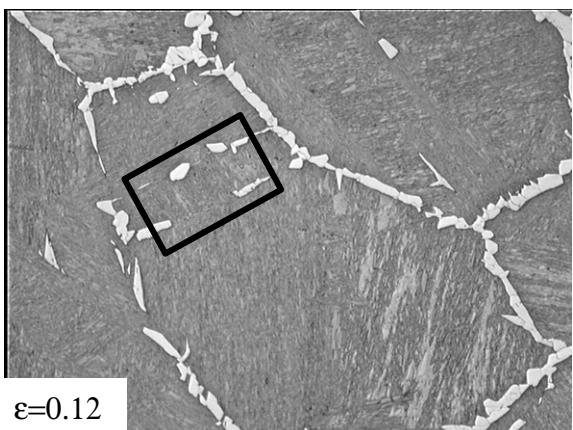
Sample strained of 40% in torsion



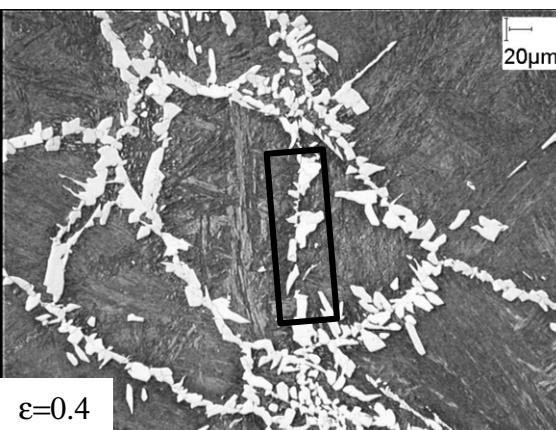
## Microstructural evolution with austenite strain level



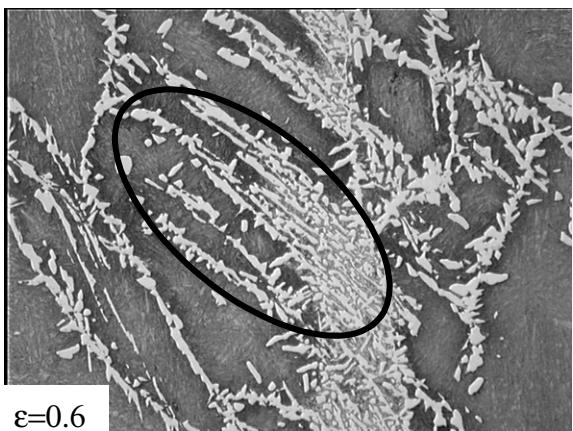
1 minute  
700 °C



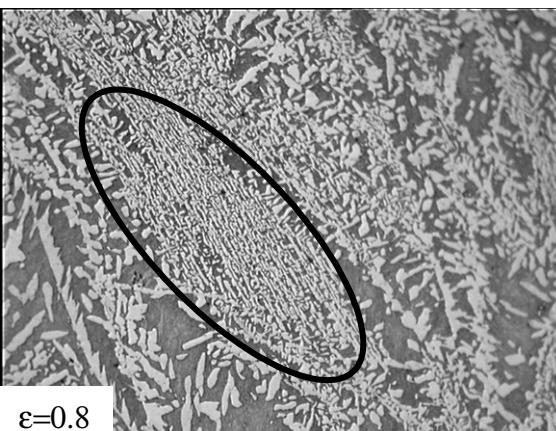
$\varepsilon=0.12$



$\varepsilon=0.4$

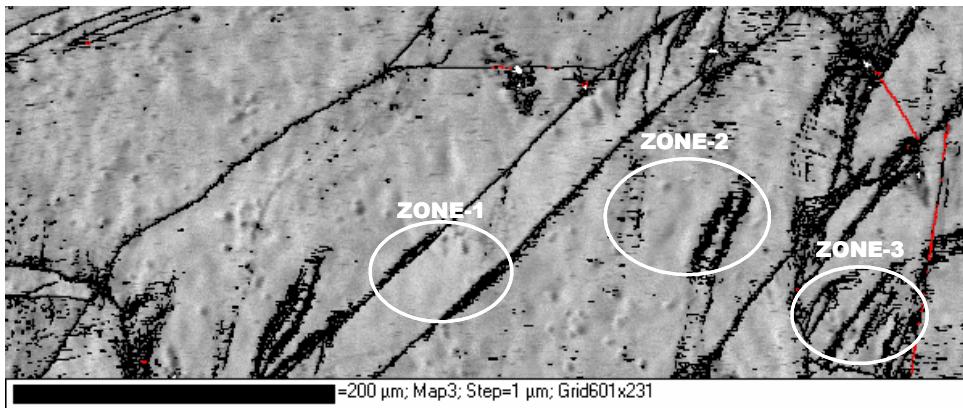


$\varepsilon=0.6$

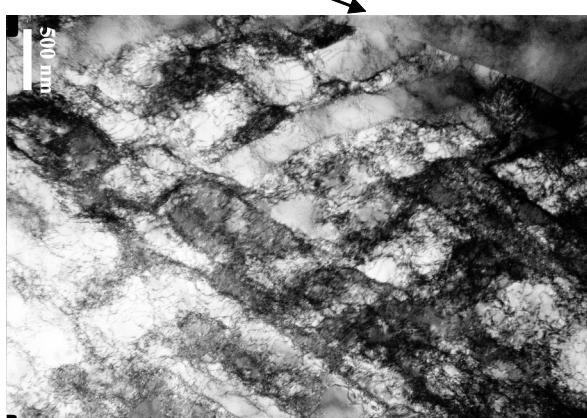
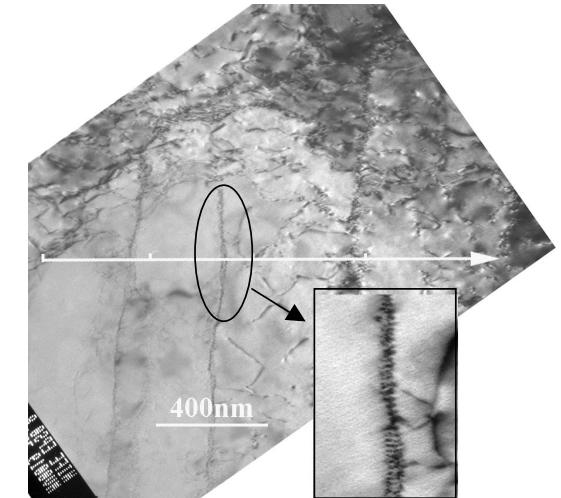


$\varepsilon=0.8$

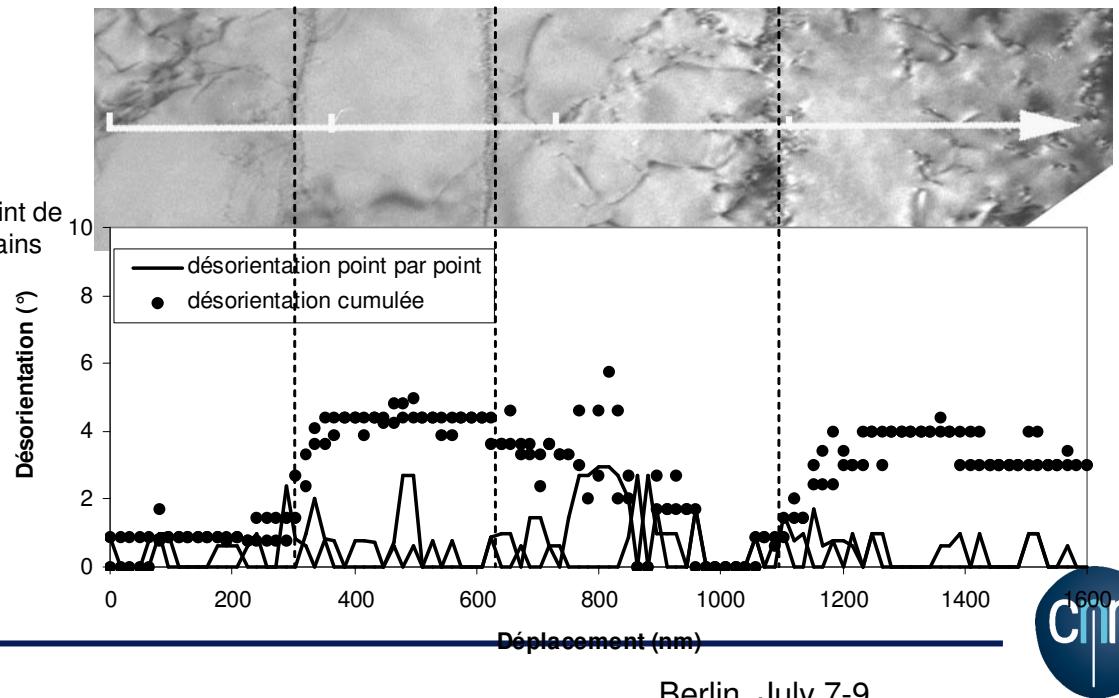
# Deformed austenite (Fe-Ni alloy), EBSD quality contrast, and TEM observations

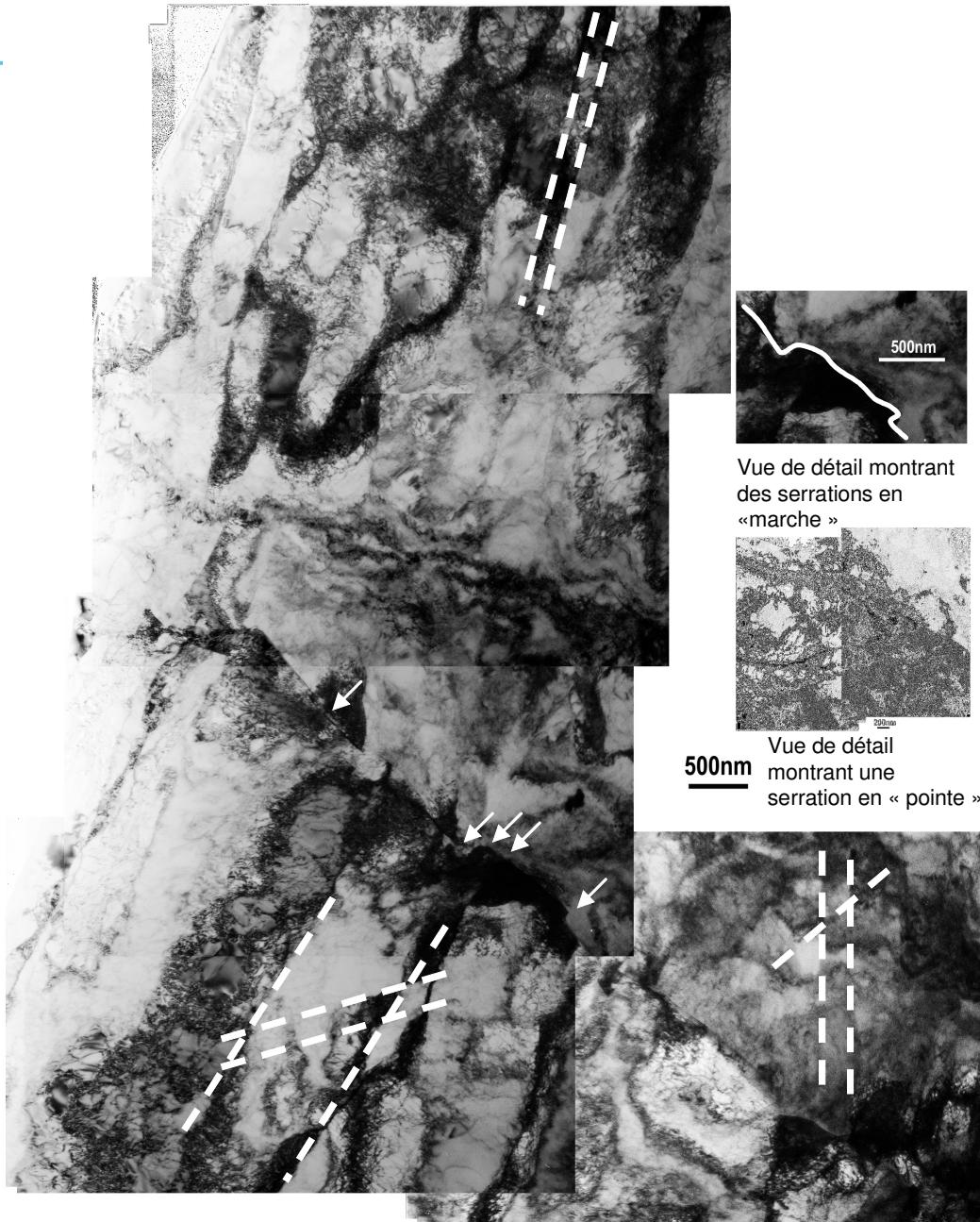


EBSD index quality contrast



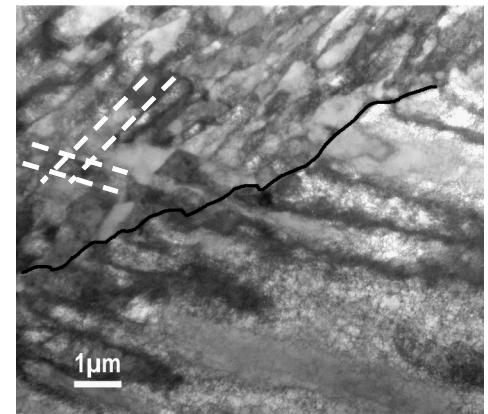
BF TEM observations





Vue de détail montrant des serrations en «marche »

Vue de détail montrant une serration en « pointe »



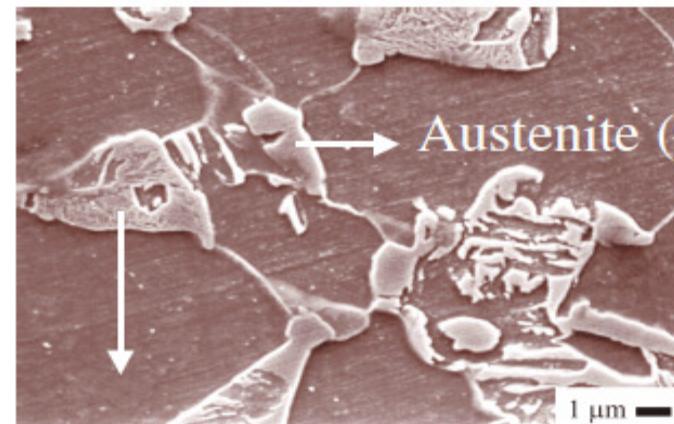
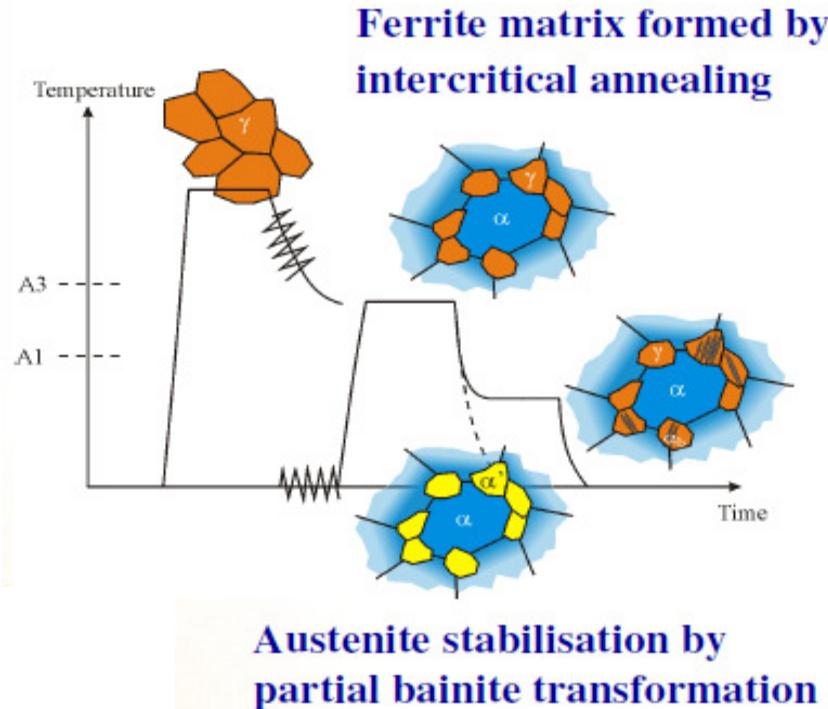
## 2) TRIP Steels

Multiphased TRIP steels, Nippon Steel, E Rauch @SIMaP

Stainless austenitic TRIP steels, M. Véron Coll. D. Embury, K. Spencer, P. Jacques

2 Familles d'acier TRIP :

a) Aciers au carbone



Martensite

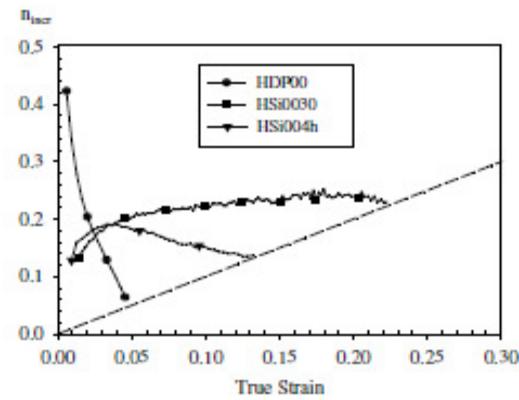
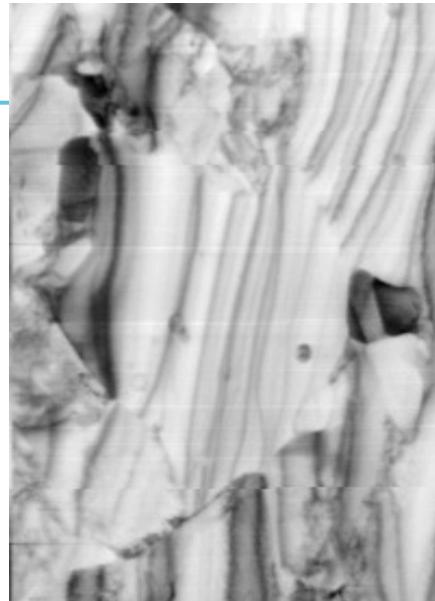




Image MET BF



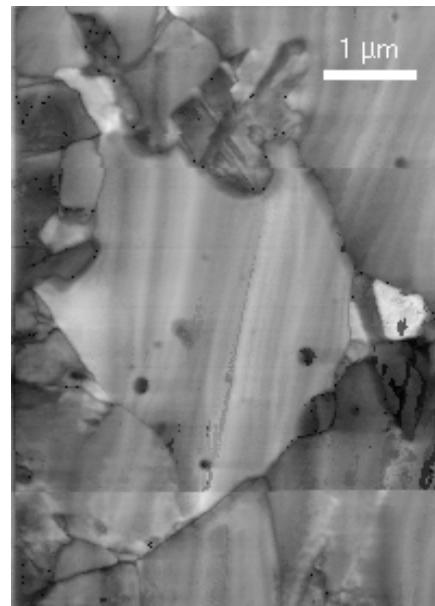
Virtual Bright Field

JEOL, 3010 LaB6, Spot  
25nm, Step 25nm

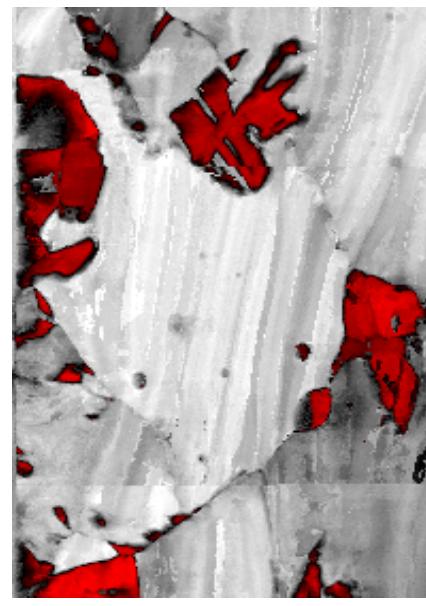


Grenoble INP

Carte d'orientation



Robustesse



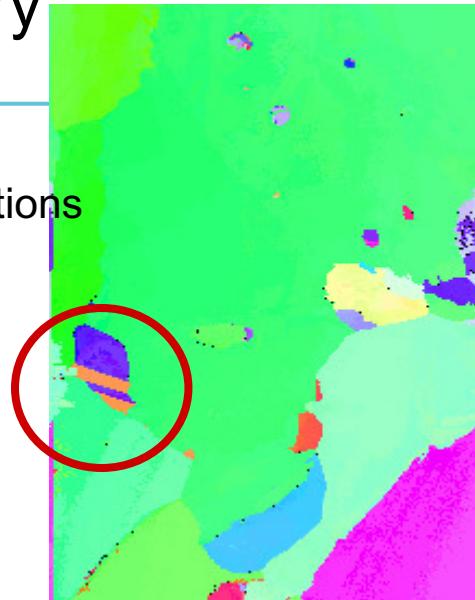
identification des phases : cfc

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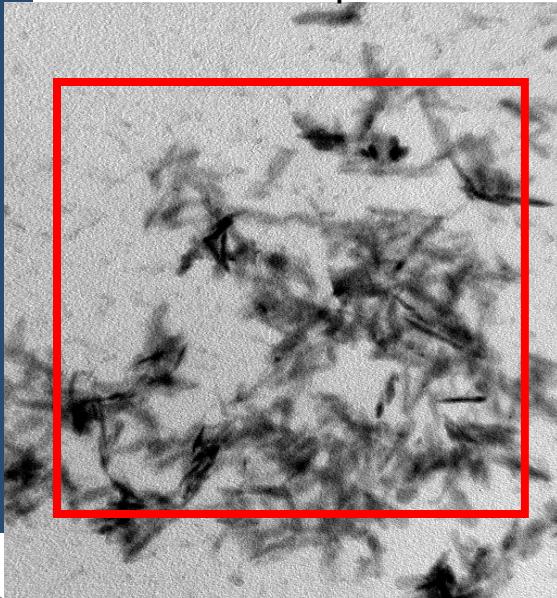


# Complementary observations

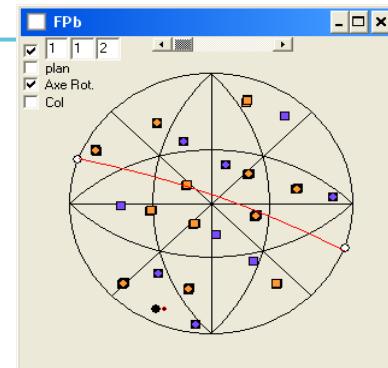
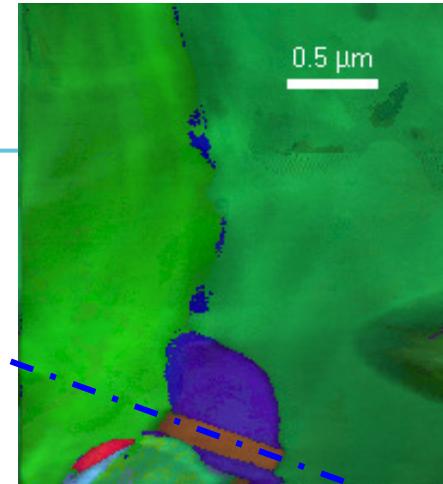
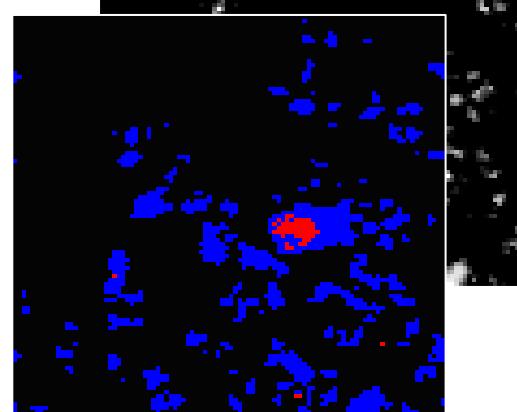
Twin observations



Precipitates identification



0.2  $\mu\text{m}$

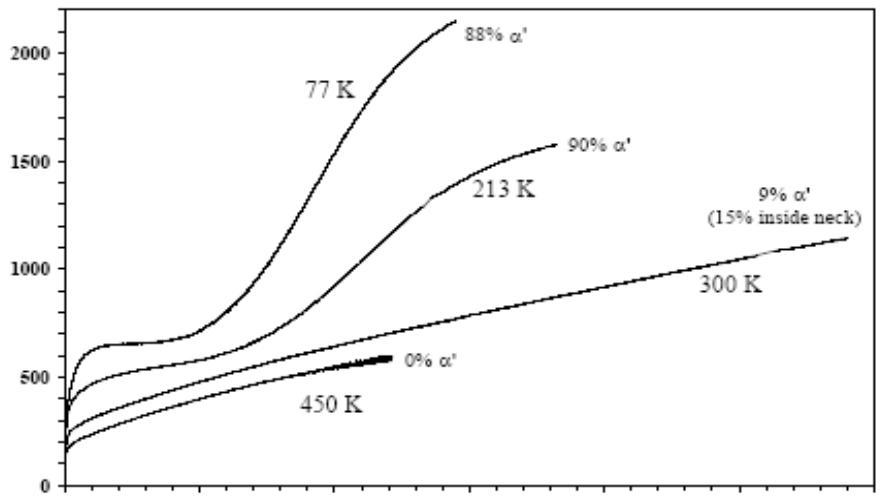


Blue : cementite

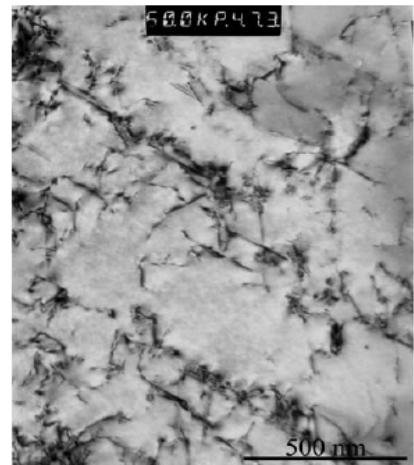
Red : TiN

## b) Austenitic stainless trip steels: deformation mechanisms

Contrainte vraie



Déformation vraie

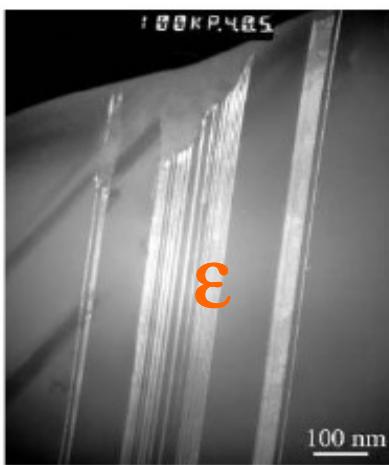


Grenoble INP

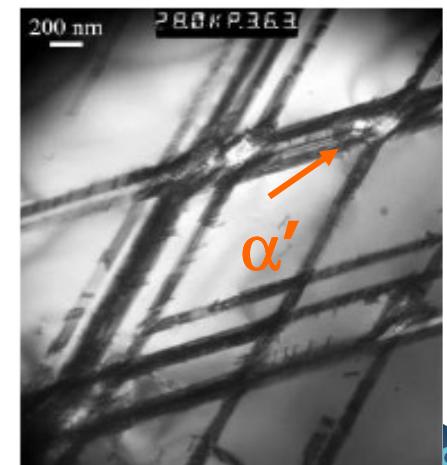
RT



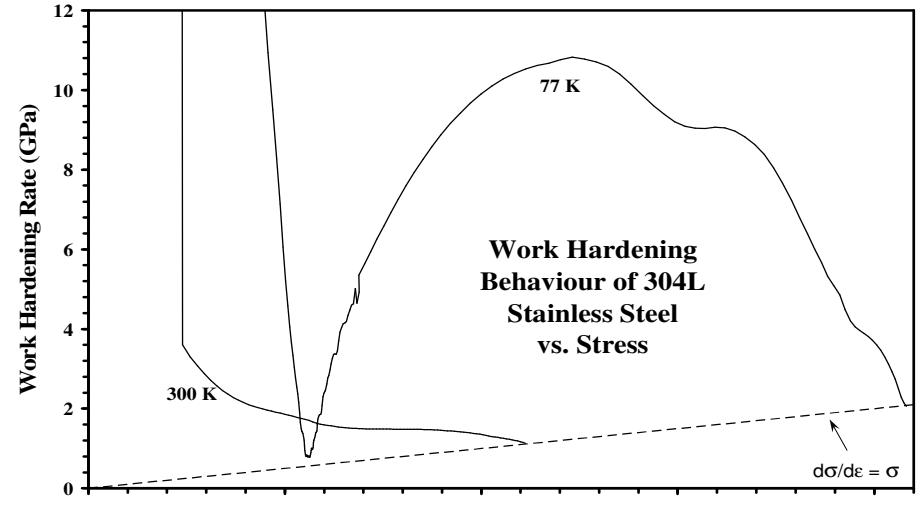
A 77K, law  $\dot{\epsilon}$



A 77K,  $\varepsilon = 5\%$



A 77K,  $\varepsilon = 10\%$

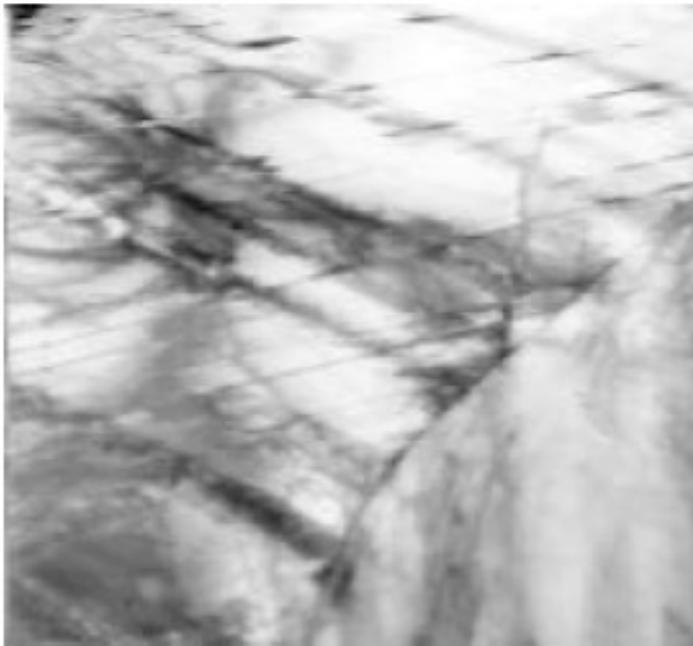


Contrainte vraie

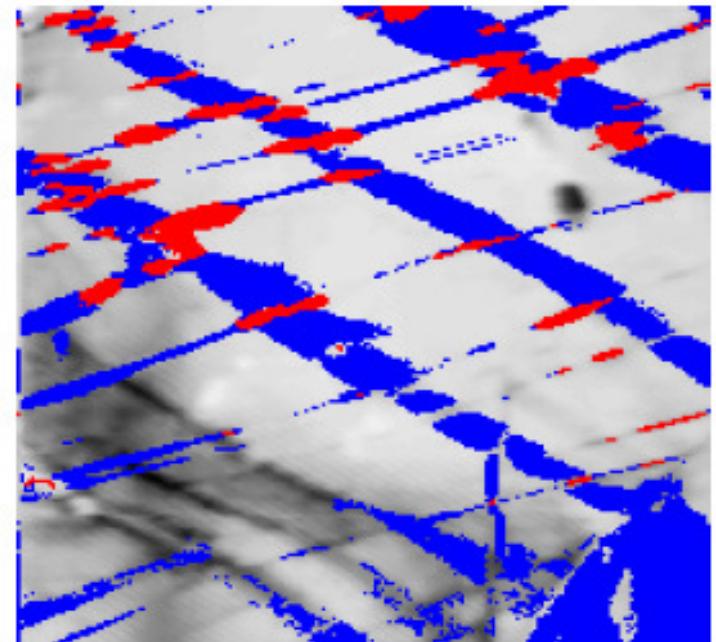
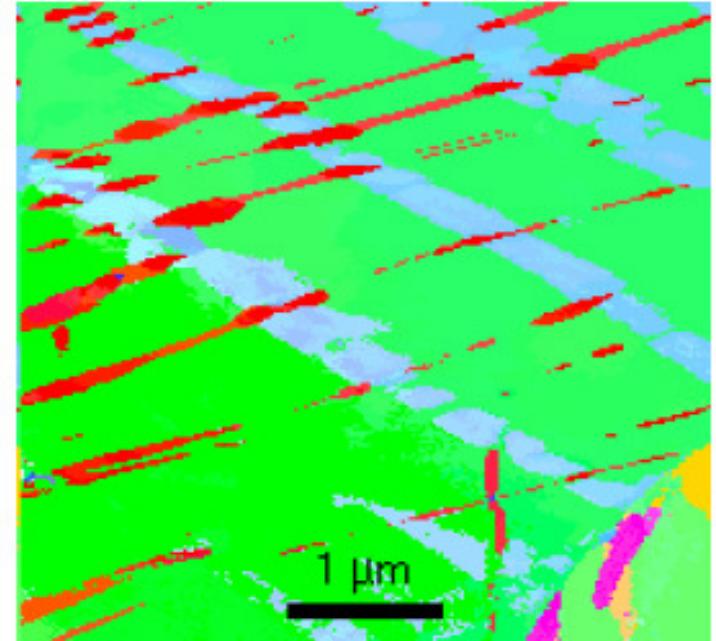
CIIIS

## Phase identification

+ orientation of phase relationships



JEOL, 3010 LaB6, Spot  
25nm, Step 25nm  
+ precession angle 0,4°

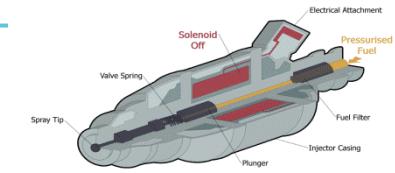


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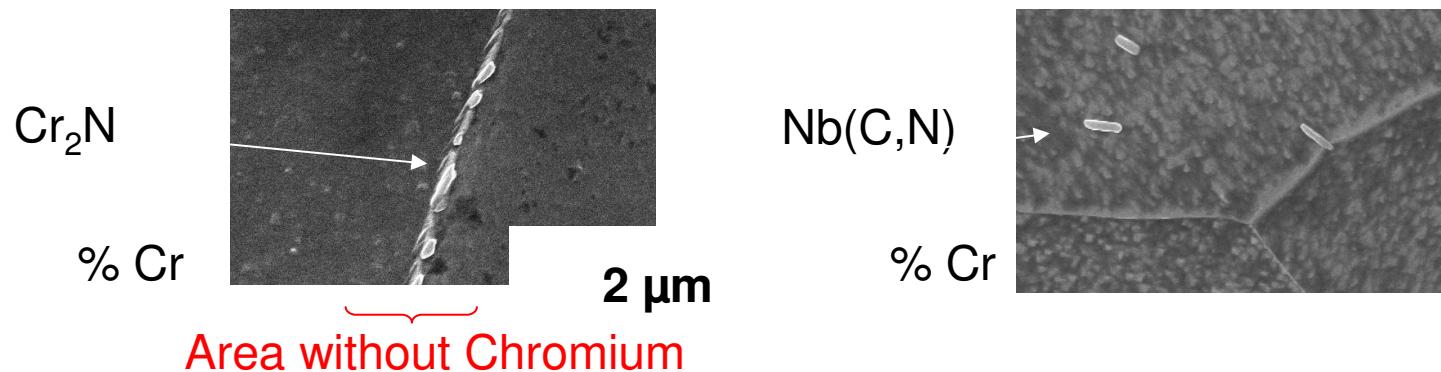
### 3) Microstructural optimization for magnetic actuators

- Alloys compositions

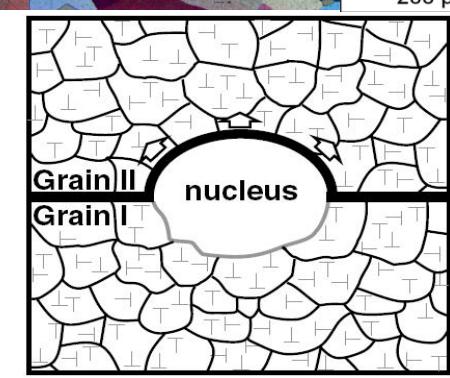
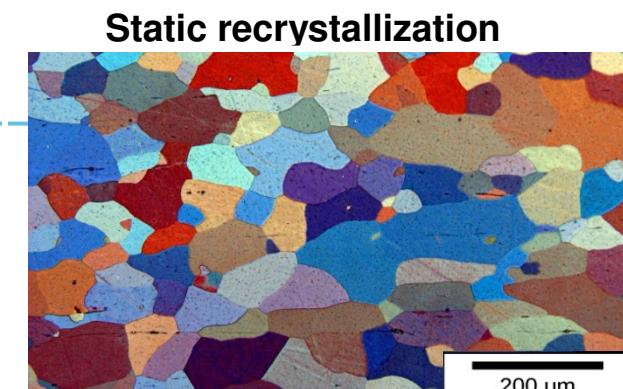
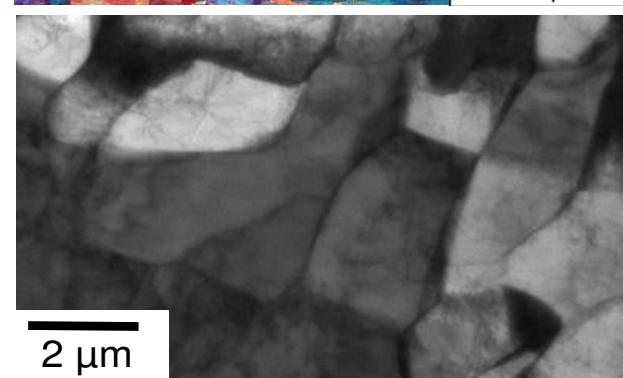
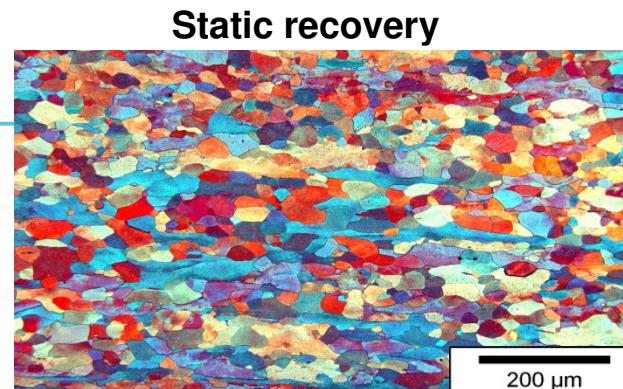
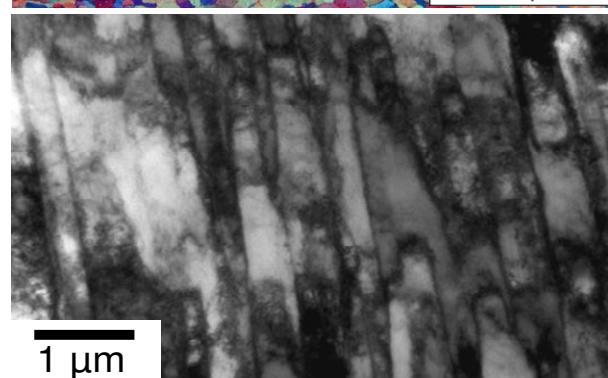
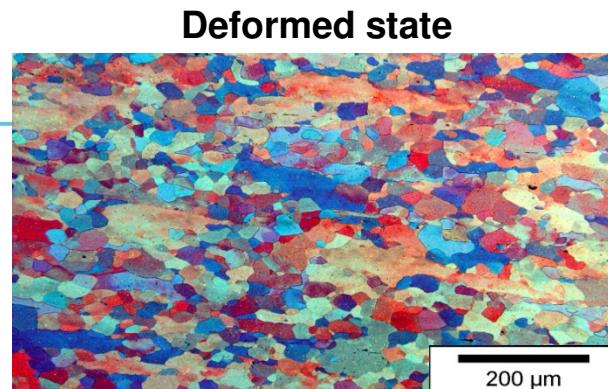
AISI	EN	C	N	Cr	Nb
430	1.4016	0.017	0.028	16.083	0.004
430Nb	1.4511	0.015	0.012	16.284	0.27



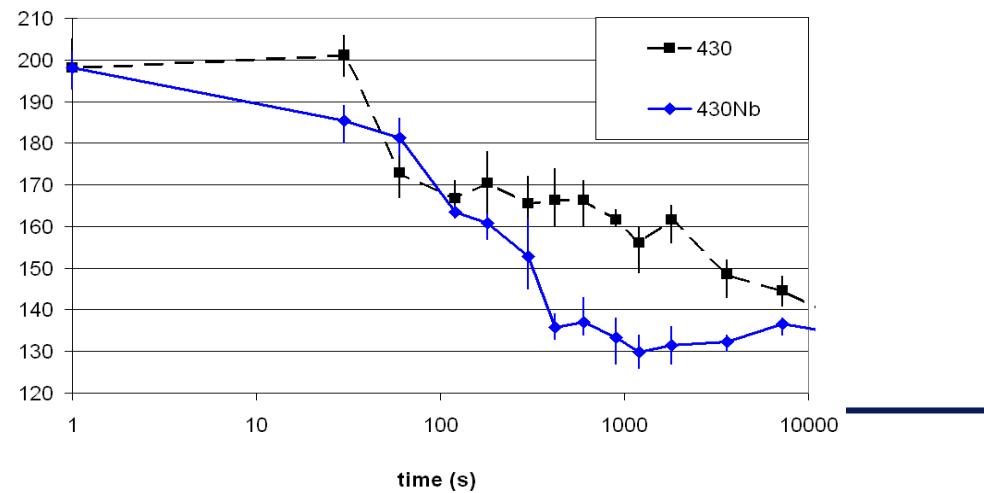
- Ferritic Stainless steel 430 ( $A_1 = 880^\circ\text{C}$ )
- Stabilized Ferritic stainless steel: 430Nb (ferritic at all T)



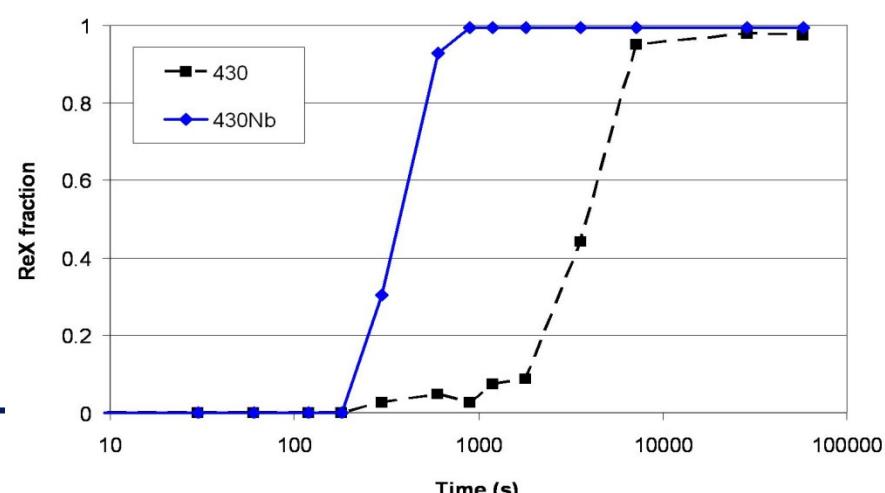
Grain scale



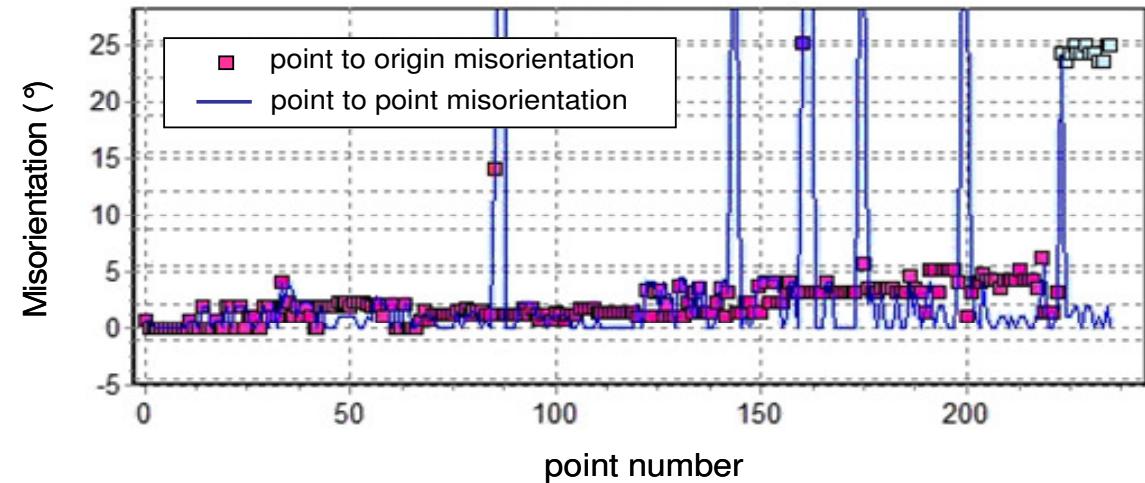
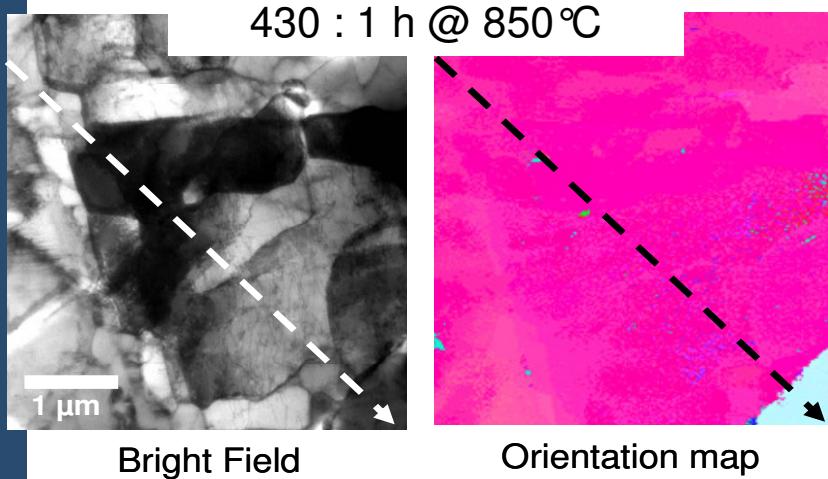
### Recovery



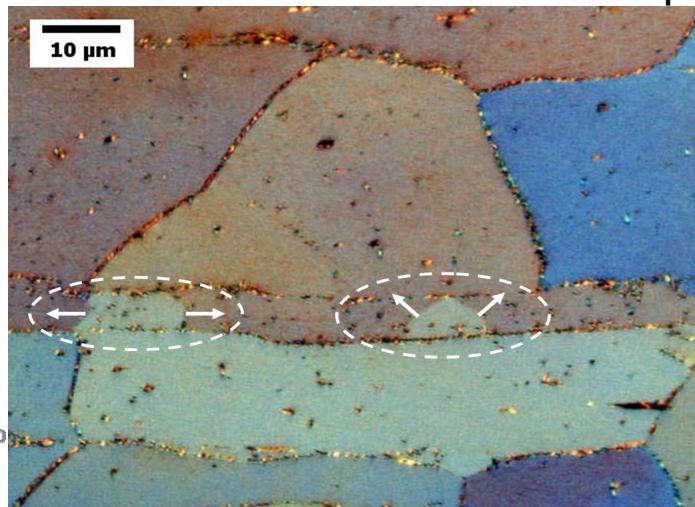
### Recrystallized fraction



- Recrystallization mechanism
  - Nucleation
- We did not observed large misorientation, even in recovered area



Complementary observation of bulging at GB's

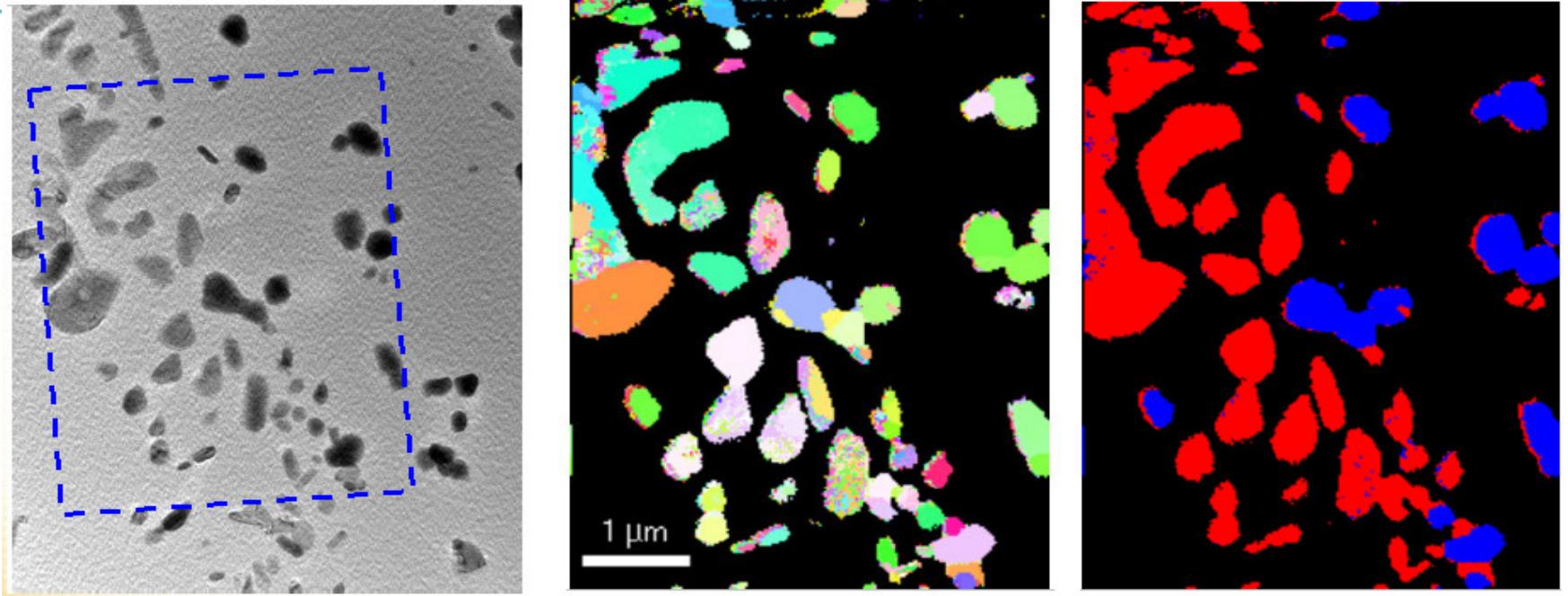


430 : 7 min @ 850 °C

Nucleation mechanisms : SIBM, typical for low strain level

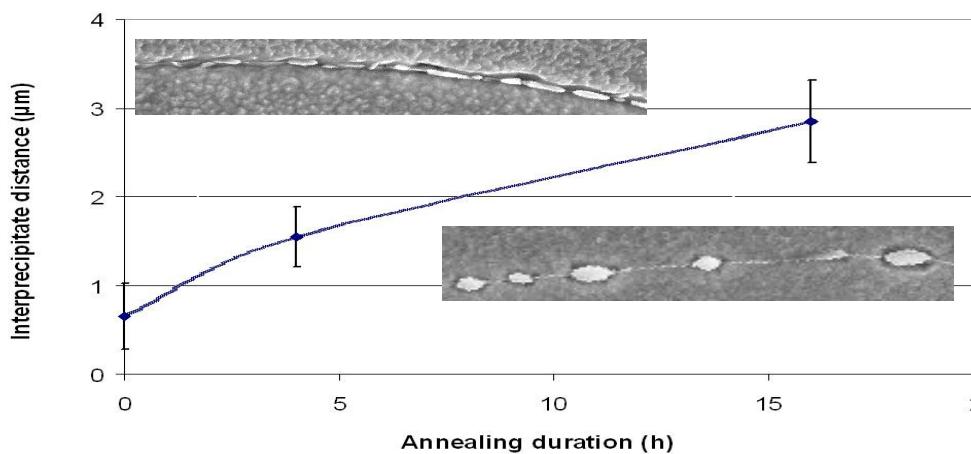


- Identification of the precipitation at the GBs (alloy 430) :



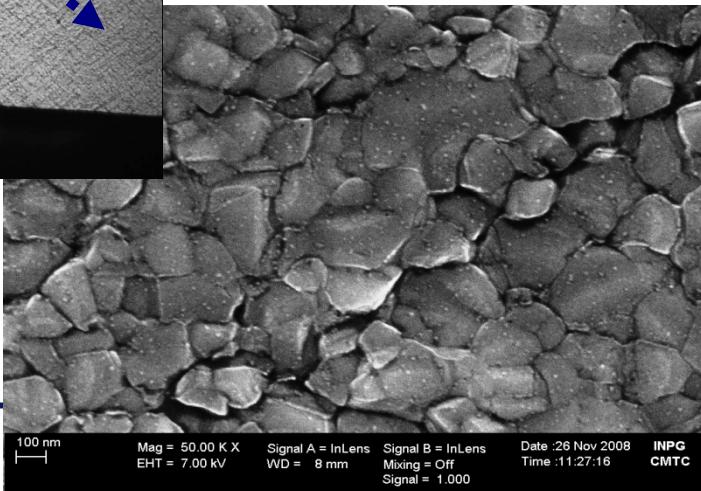
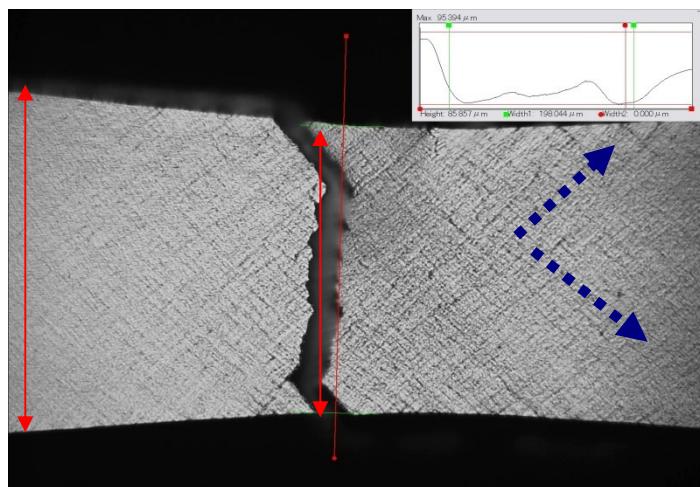
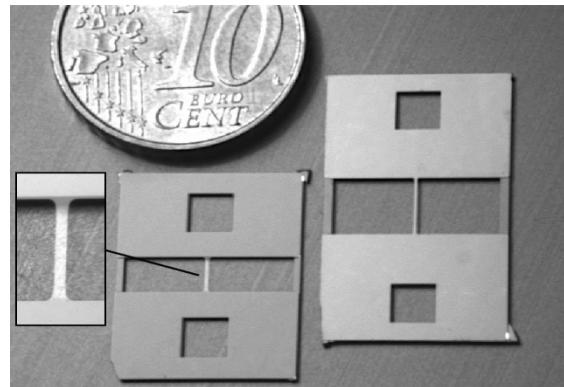
Phase identification: carbides (blue), and nitrides (red)

Recrystallization starts  
when precipitates have  
coarsened

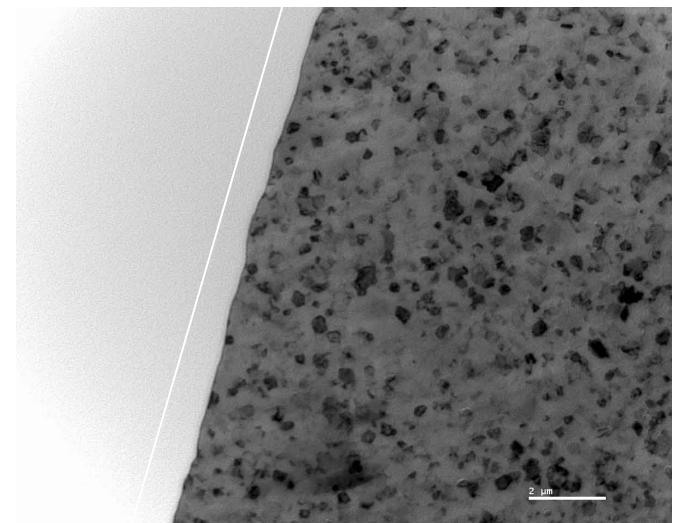


## 4) Thin Films

## 4) Thin Films : S. lay, Grenoble

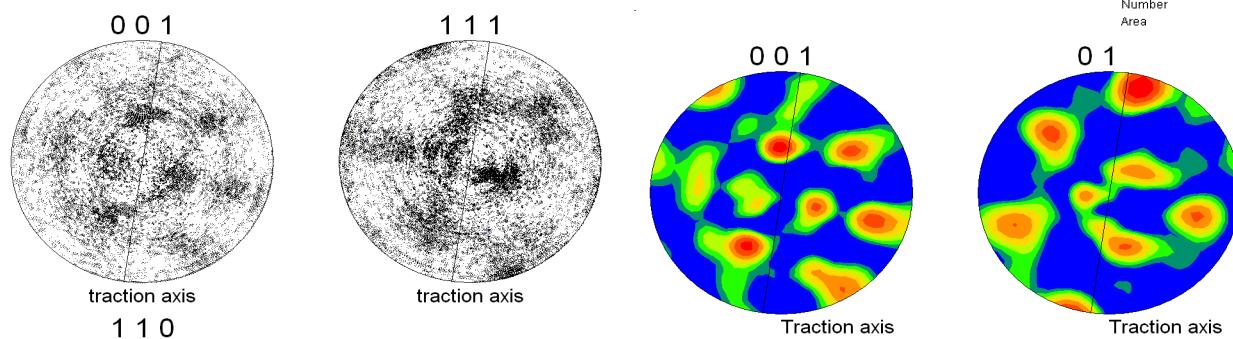
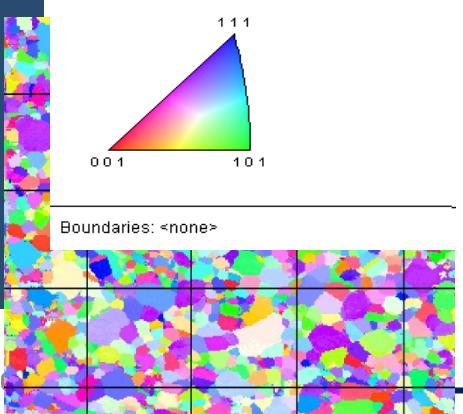
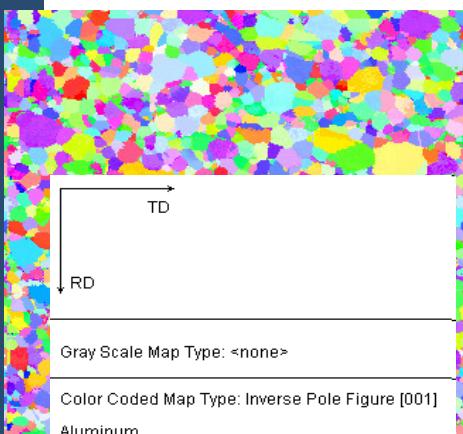
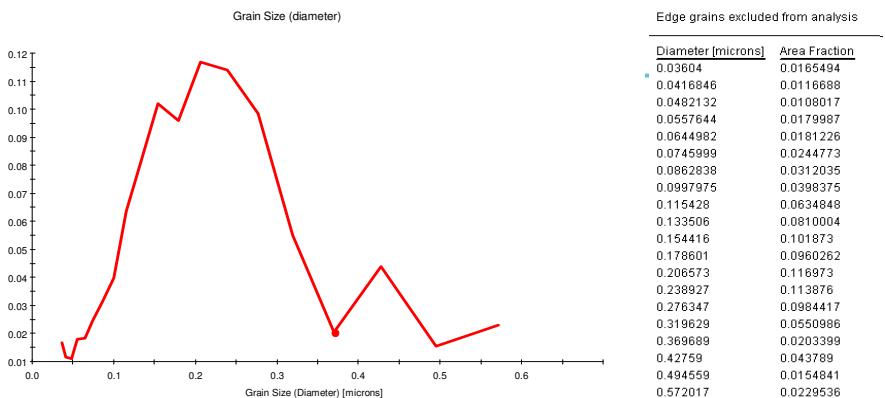
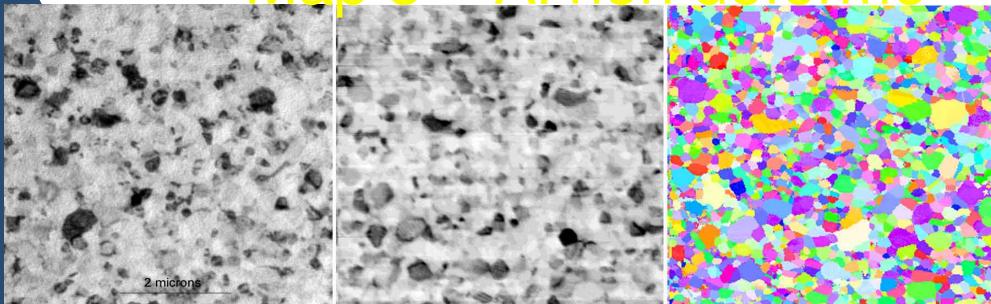


Non déformé

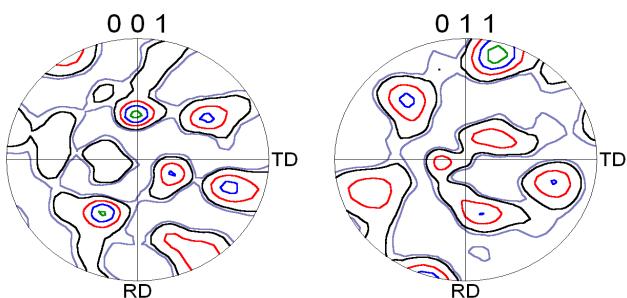
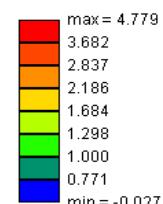


déformé  
Berlin, July 7-9

## Map 3 – Al non déformé



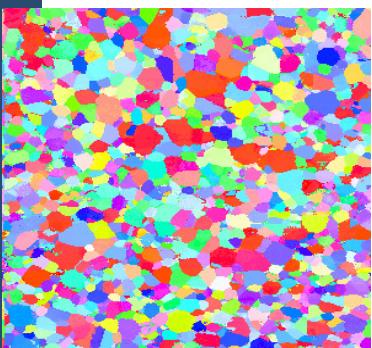
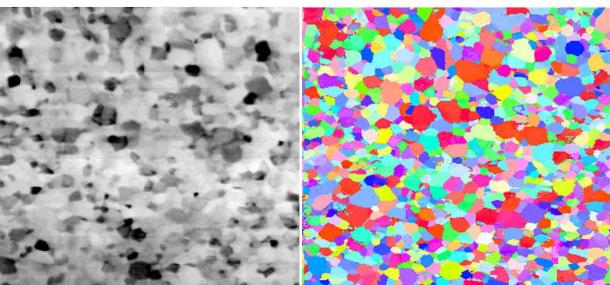
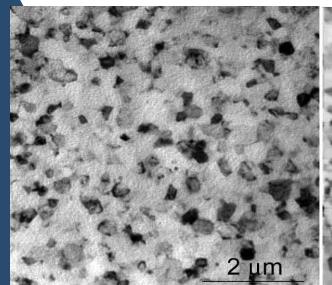
Texture Name: Harmonic: L=16, HW=5.0  
Calculation Method: Harmonic Series Expansion  
Series Rank (l): 16  
Gaussian Smoothing: 5.0°  
Sample Symmetry: Triclinic



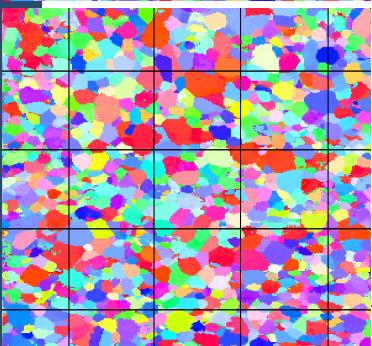
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# Map 3 Al déformé



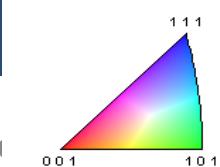
ACOM



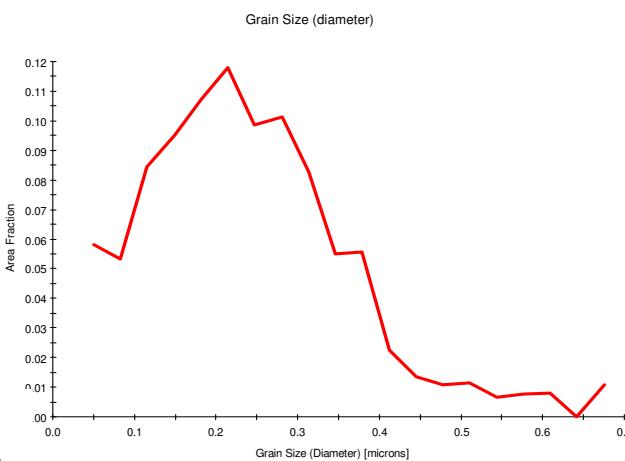
OIM

Color Coded Map Type: Inverse Pole Figure [001]

Aluminum



Boundaries: <none>



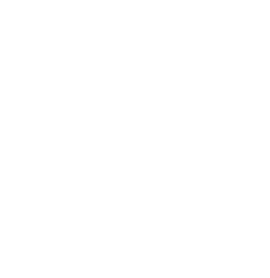
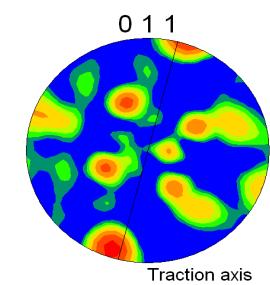
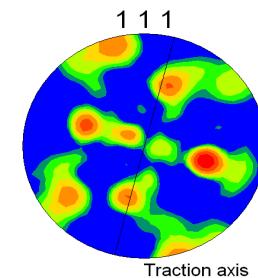
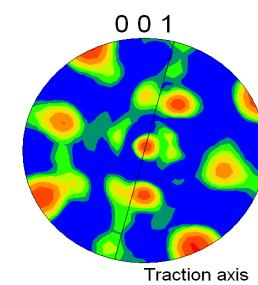
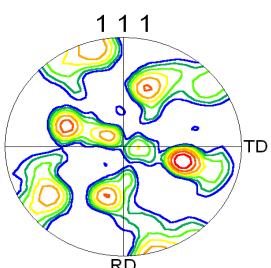
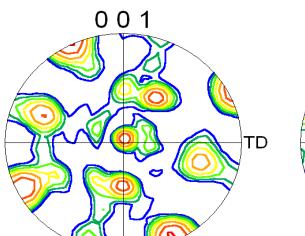
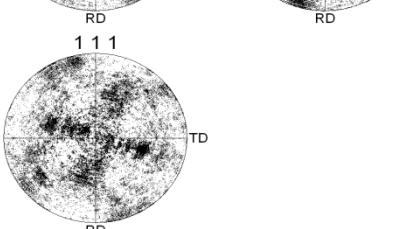
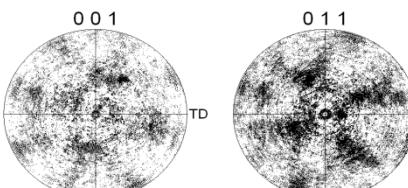
Edge grains excluded from analysis

Diameter [microns]	Area Fraction
0.0499649	0.057976
0.0828725	0.0531521
0.11578	0.084316
0.148688	0.0952016
0.181595	0.107325
0.214503	0.11807
0.24741	0.0984303
0.280318	0.1012
0.313225	0.0825549
0.346133	0.0549387
0.379041	0.0555768
0.411948	0.0225625
0.444856	0.0135528
0.477763	0.0106687
0.510671	0.0114599
0.543578	0.00653395
0.576486	0.00749107
0.609393	0.00811639
0.642301	0
0.675209	0.0108729

Average  
Number  
Area

0.0979333

0.241055

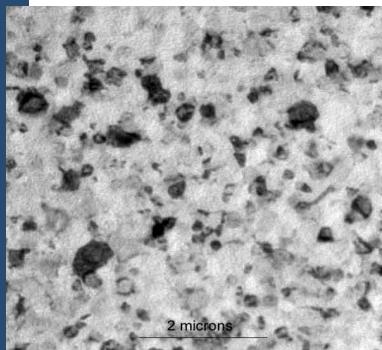


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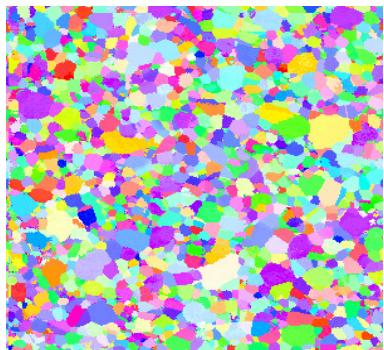


## Aluminium before deformation

TEM image



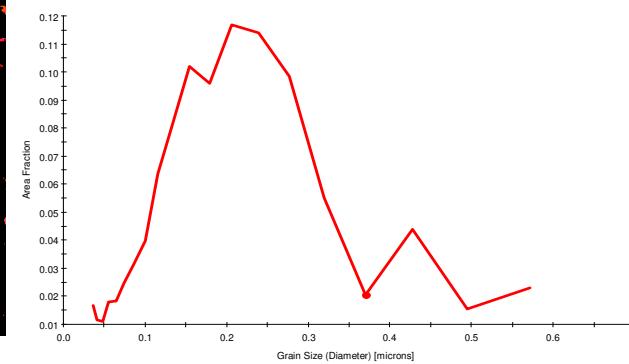
Orientation map



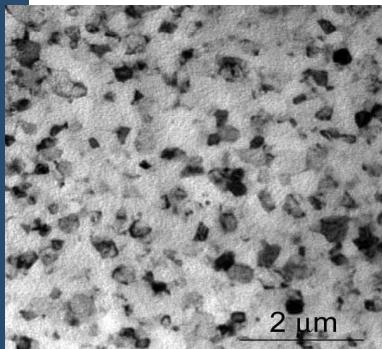
Grains close to <001> direction



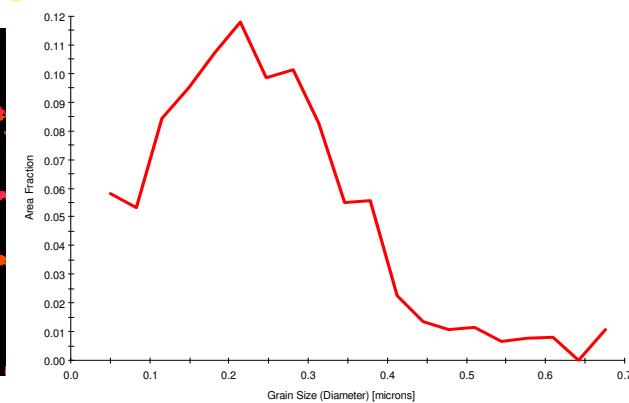
Grain size distribution



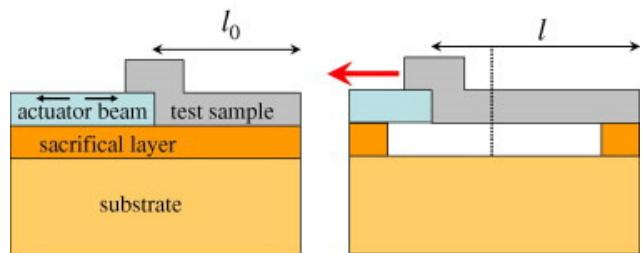
## Aluminium after deformation



Grain Size (diameter)



## 4) Thin Films : S. Godet, Bruxelles

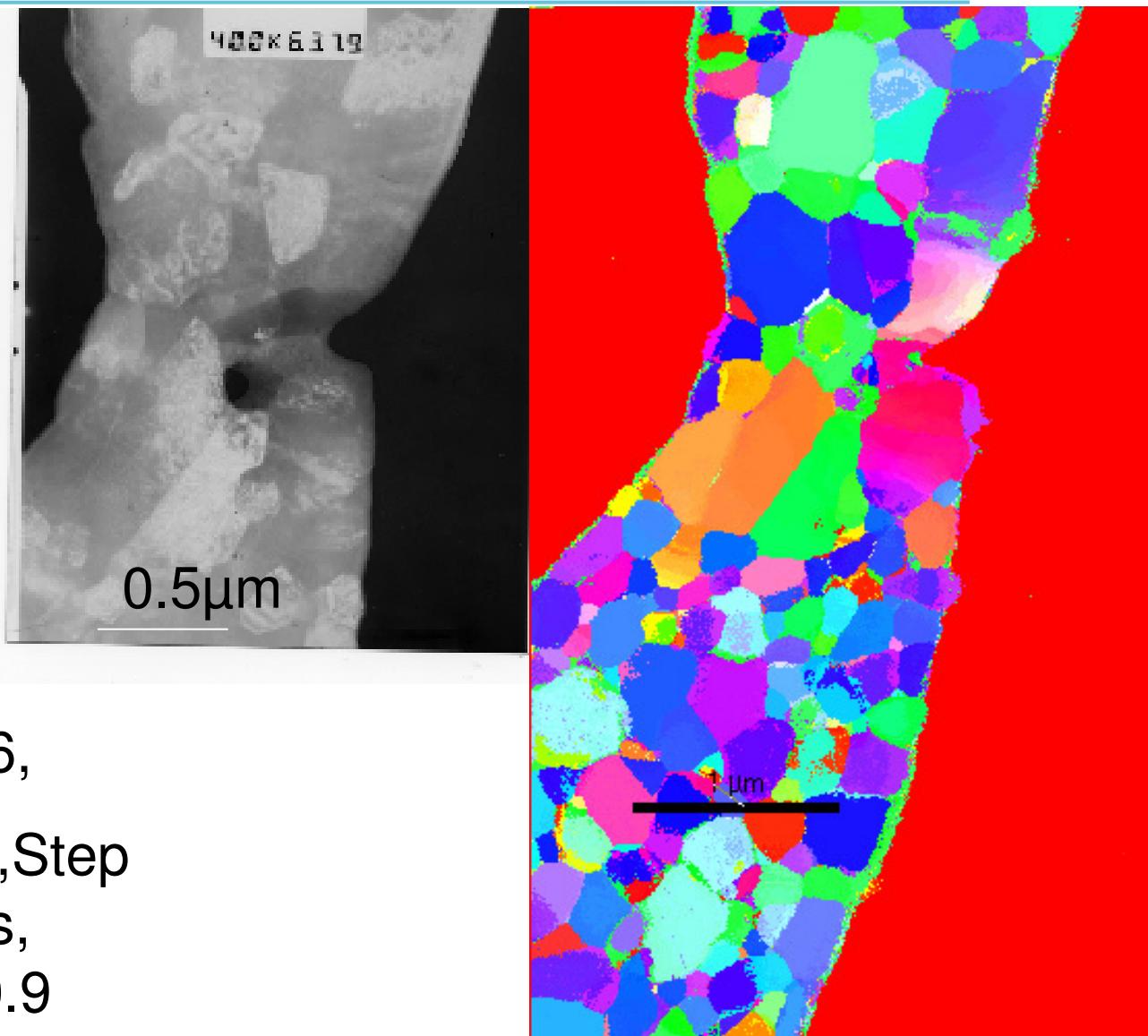


$$\varepsilon^{mech} = \ln\left(1 + \frac{\Delta l}{l_0}\right) + (\alpha_{sub} - \alpha)\Delta T$$

$$\varepsilon_{act}^{mech} = \ln\left(\frac{l_{0act} - \Delta l}{l_{0act} - \Delta l_{free}}\right)$$

$$\sigma_{act} = E_{act} \varepsilon_{act}^{mech}$$

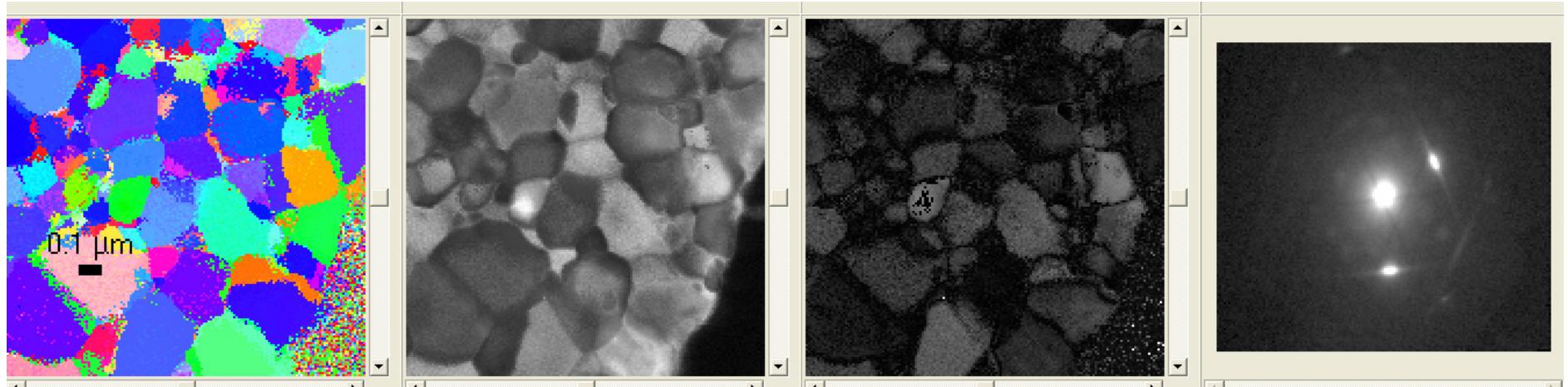
$$\sigma = \sigma_{act} \frac{S_{act}}{S}$$



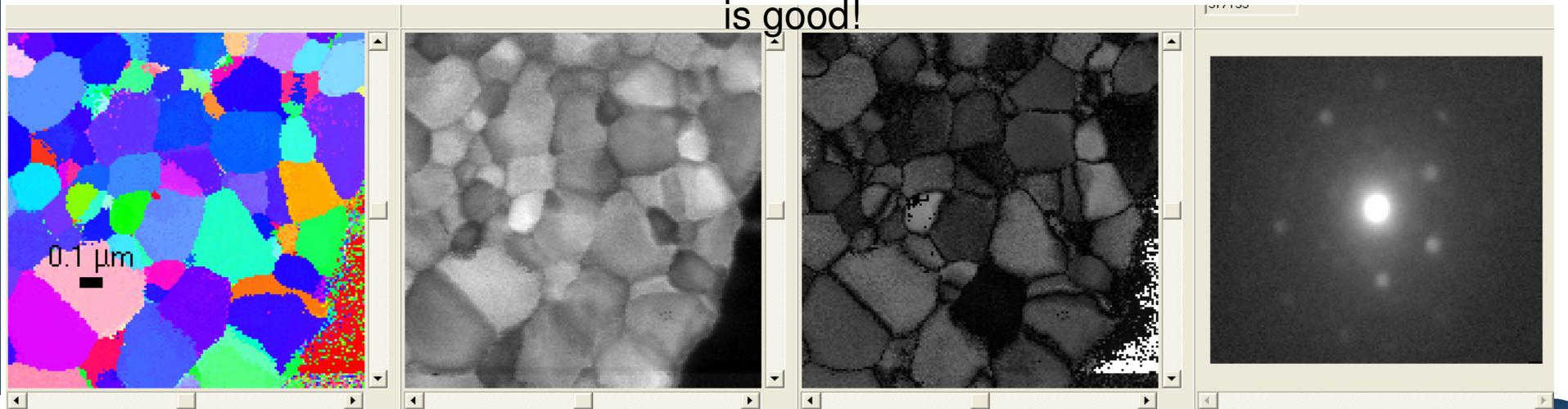
CM20, LaB6,

Spot size 10nm, Step  
10nm, 50fps,  
precession 0.9

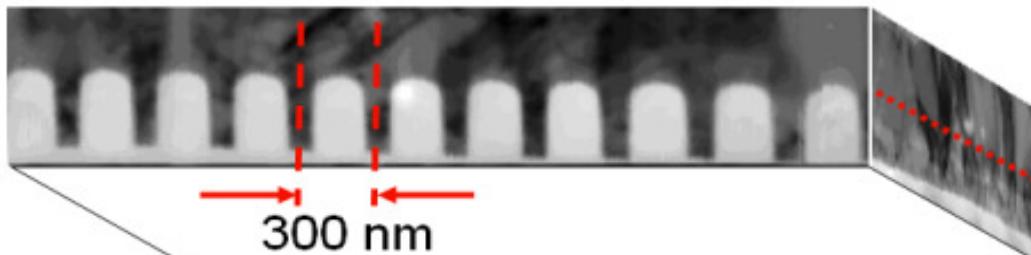
Good results were obtained with a precession angle  $\alpha = 0.9^\circ$



Thick sample, same area, diffraction patterns with kikuchi lines. Without precession, quality is poor, with precession  $\alpha = 0.9^\circ$ , diffraction patterns are « cleaned », and indexing is good!



# Tailles de grains et désorientation dans des lignes de Cu de 80nm



500x100 steps (6.5 nm each), Spot size 25 nm  
Scanning time : 19 min (44 fps)

Side view

## CROSS VIEW (orientation map)

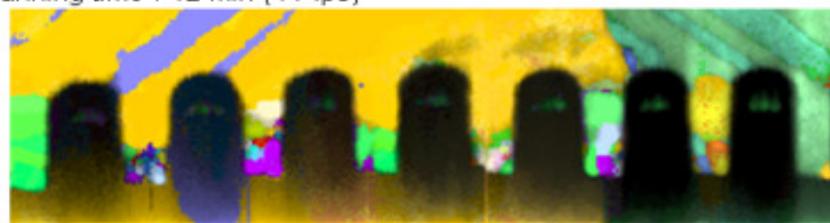
Grain size of the order of 30 nm may be identify despite the use of a conventional LaB<sub>6</sub> equipped Jeol 3010 TEM (spot size 25 nm)



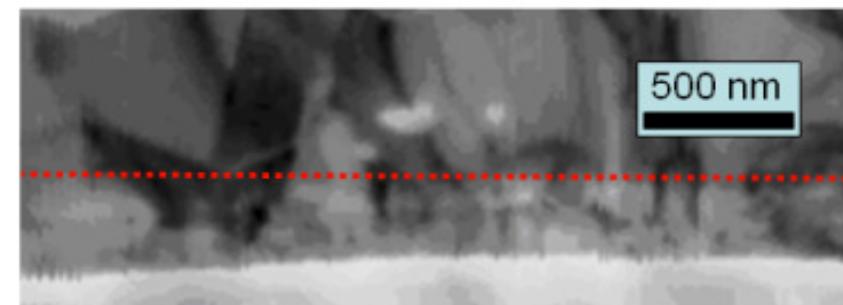
250x100 steps (13 nm each)  
LaB<sub>6</sub> equipped Jeol 3010 TEM (spot size 25 nm)  
Scanning time : 10 min (44 fps)



300x100 steps (6.5 nm each), spot size 15 nm  
Scanning time : 12 min (44 fps)



SIDE VIEW (orientation and index superimposed map)  
The two scans were performed with different settings  
They demonstrated the reproducibility of the identification

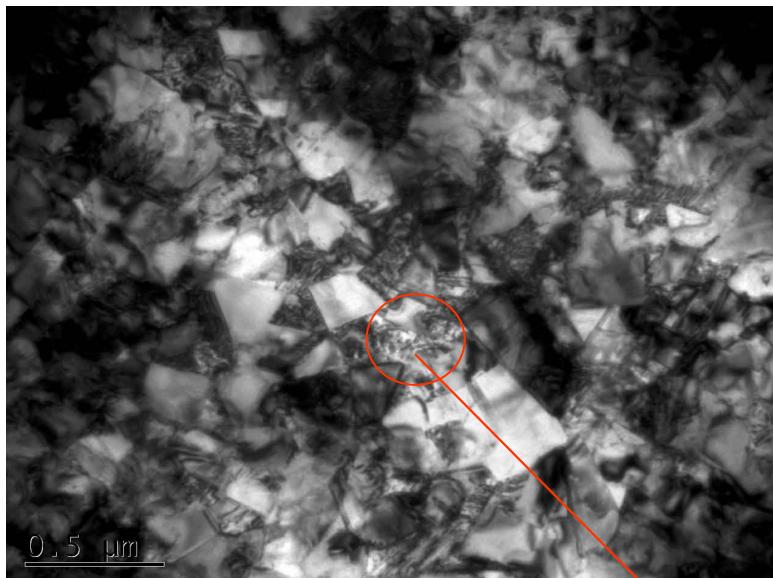


## Credits:

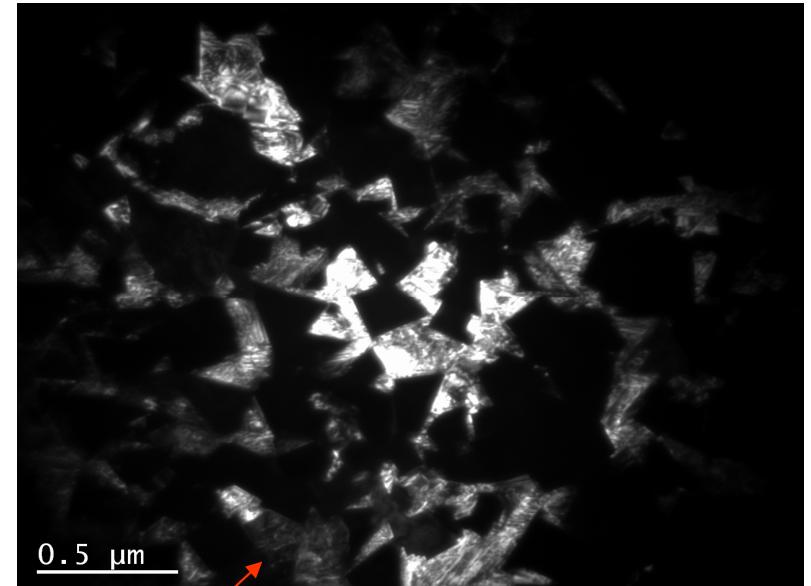
- Stefan RÖDANDETTER - CIMaD - Grenoble INP

## 5. Identification des phases et des orientations dans les matériaux frittés WC-Co

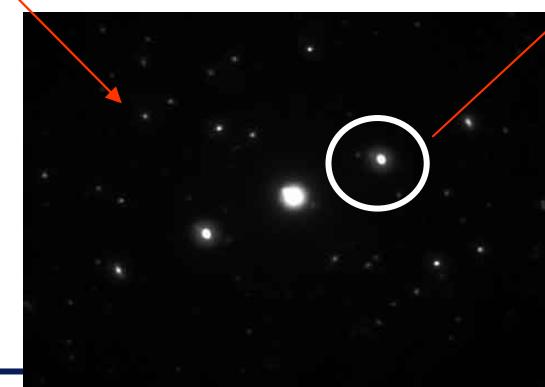
S. Lay @SIMaP



Bright field image



Dark field image : same orientation for all Co pools (Co in bright)



DP of Co

