

# LIEN SÉGRÉGATION / RECRISTALLISATION DANS LES ALLIAGES FE-SI-SN

**Myriam DUMONT**, MdC 33<sup>ème</sup> section

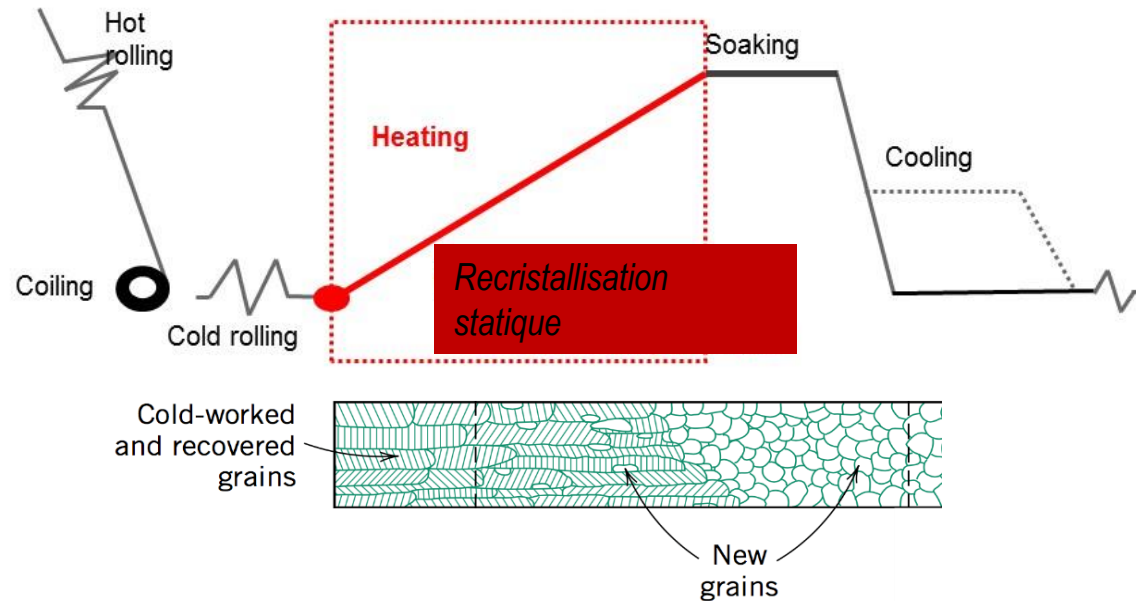
Laboratoire MSMP, Lille

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# Recristallisation statique dans les aciers

## Introduction

Route métallurgique :

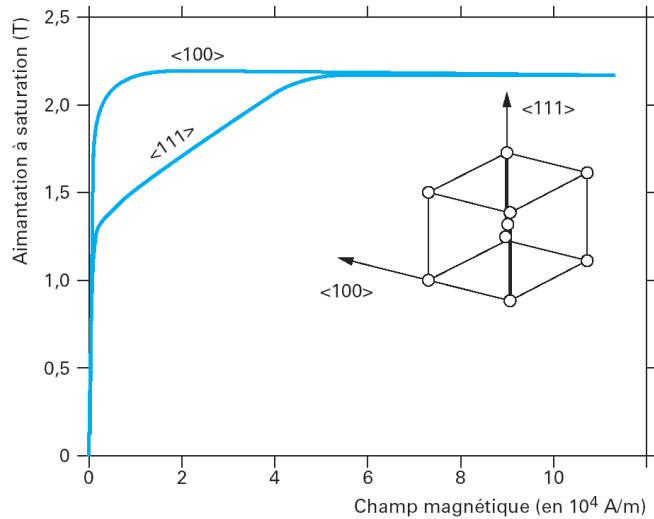


Éléments pouvant influencer la cinétique de recristallisation :

- ✓ Température de recuit
- ✓ Vitesse de chauffe
- ✓ Chimie de l'alliage :
  - Par effet de trainage des solutés pour les éléments en solution solide (solute drag)
  - Par effet d'ancrage par les précipités (Zener pinning)

# Texture et aciers électriques

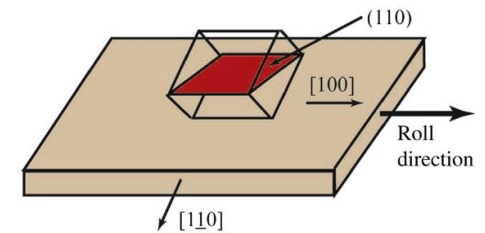
## Anisotropie magnétocristalline



Dans le fer cubique centré (ferrite)

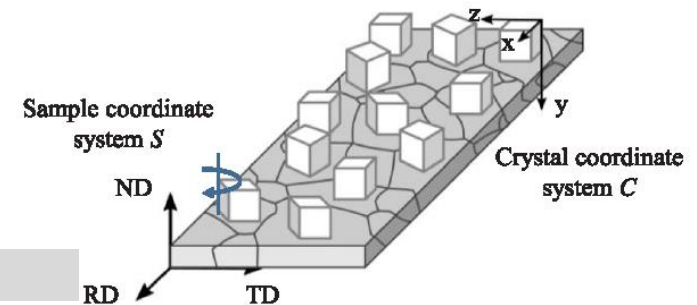
<100> : direction de facile aimantation

<111> : direction de difficile aimantation



Aciers à grains orientés (GOES) : Goss texture  $\{110\} \langle 001 \rangle$

Aciers à grains non orientés (NOES) : idéalement composante de type ND Rotated-cube  $\{100\} \langle hkl \rangle$  (ou fibre  $\Theta$ )



(b) ND-rotated cube fiber.

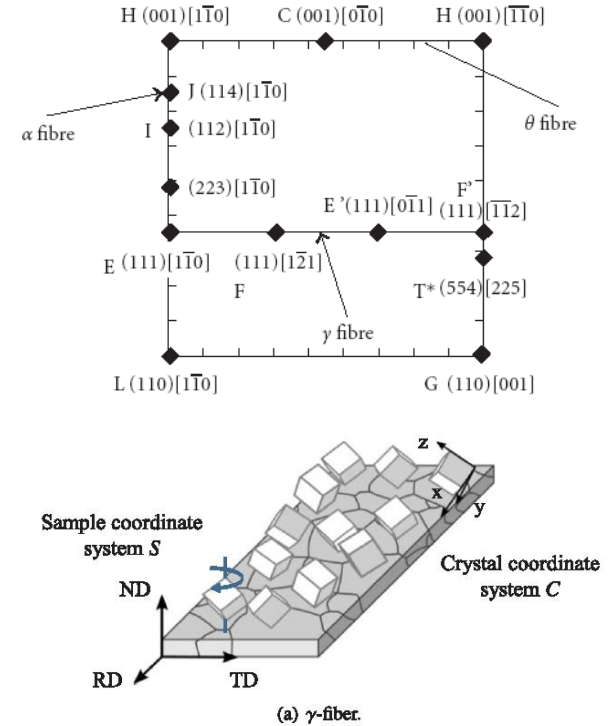
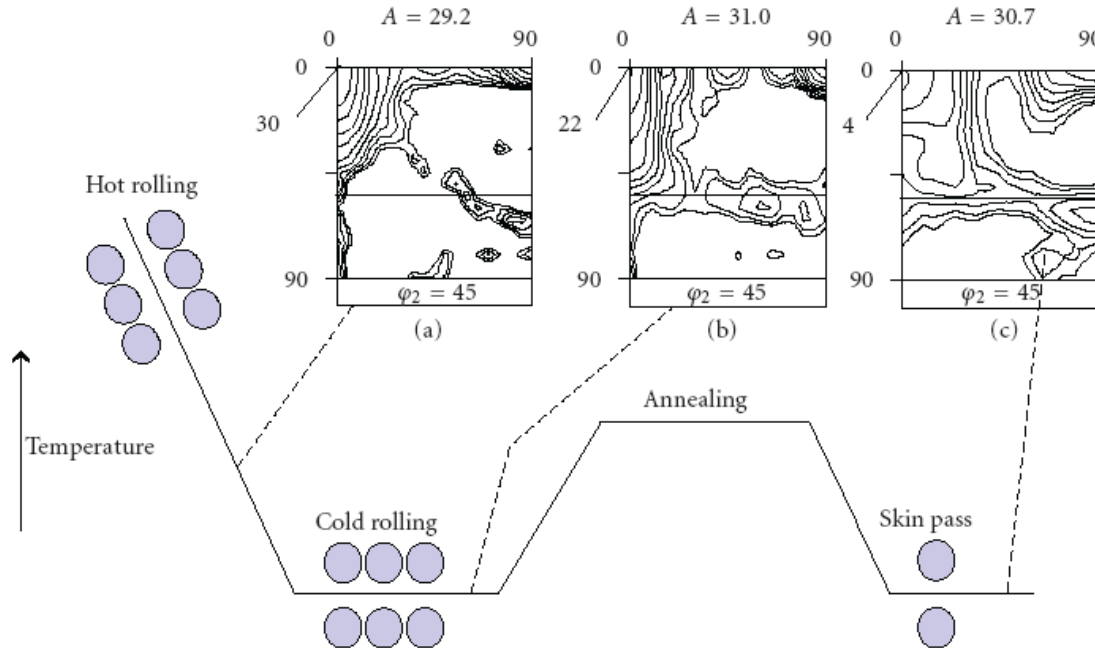
*Techniques de l'ingénieur, Matériaux magnétiques doux cristallins, T. Waeckerlé, 2010, D2121 V1*

*N. Leuning et al., On the correlation of crystallographic macro-texture and magnetic magnetization anisotropy in non-oriented electrical steels, J. of Magnetism and Magnetic Materials, Vol 490 (2019) 165485.*

# Textures de recristallisation

## Textures classiques des aciers NOES

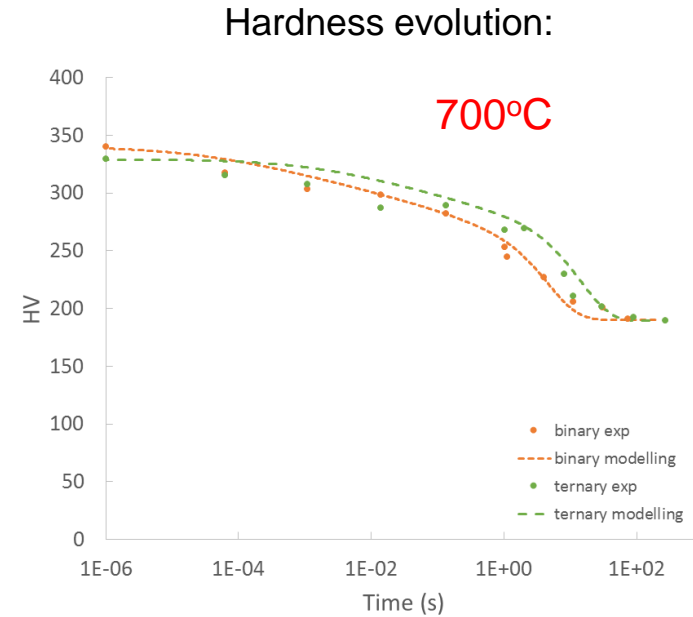
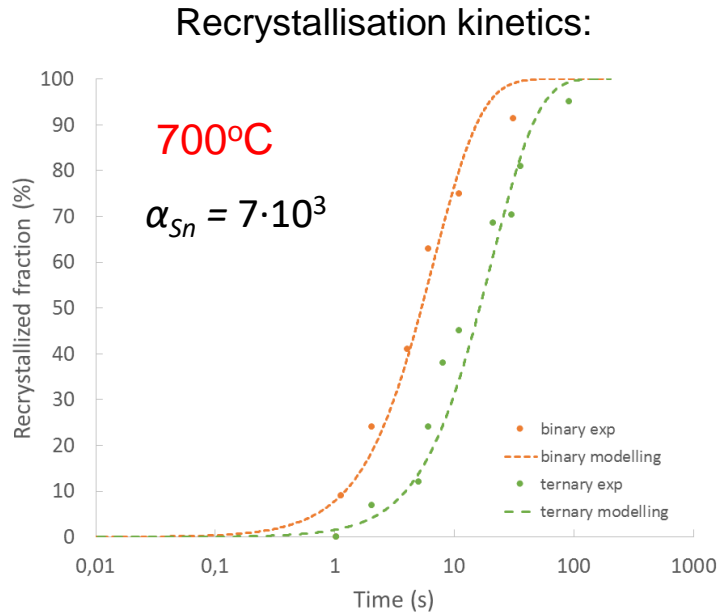
Evolution de la texture des aciers à grains non orientés suivant le procédé conventionnel



Optimisation de la texture pour réduire la composante  $\gamma$  et augmenter la composante de type ND Rotated-cube  $\{100\}\langle hkl \rangle$  (ou fibre  $\Theta$ )

L. Kestens et al., *Texture control during the manufacturing of non oriented electrical steels*, *Texture, Stress and Microstructure*, Vol 2008 172083.



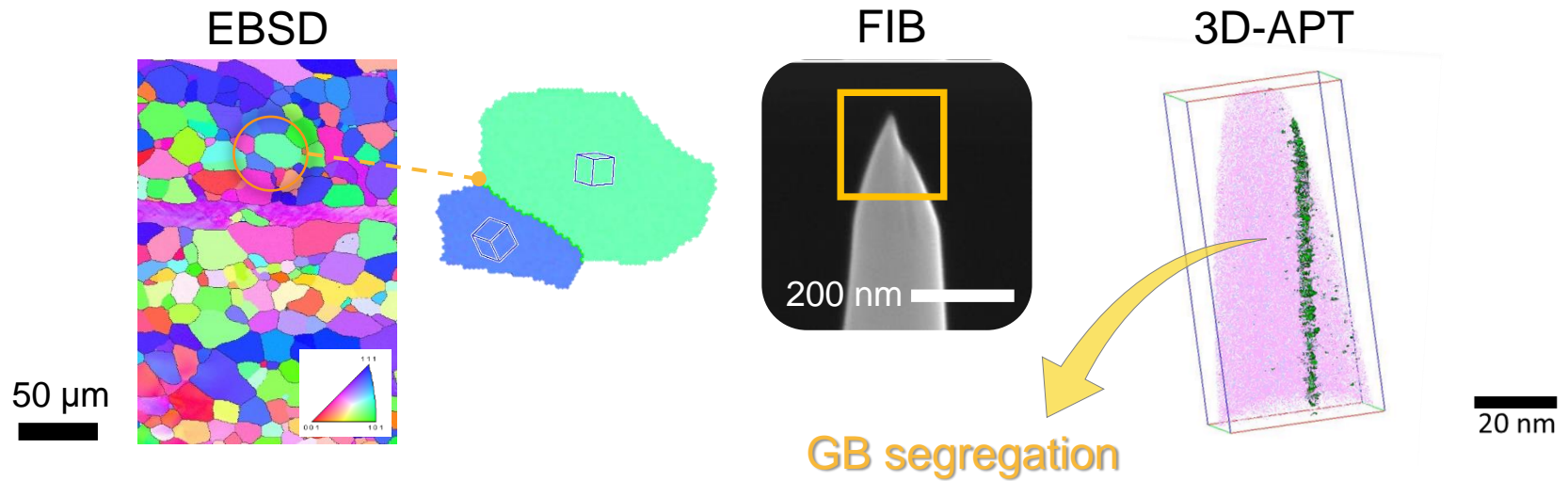


Retardation effect via solute drag.



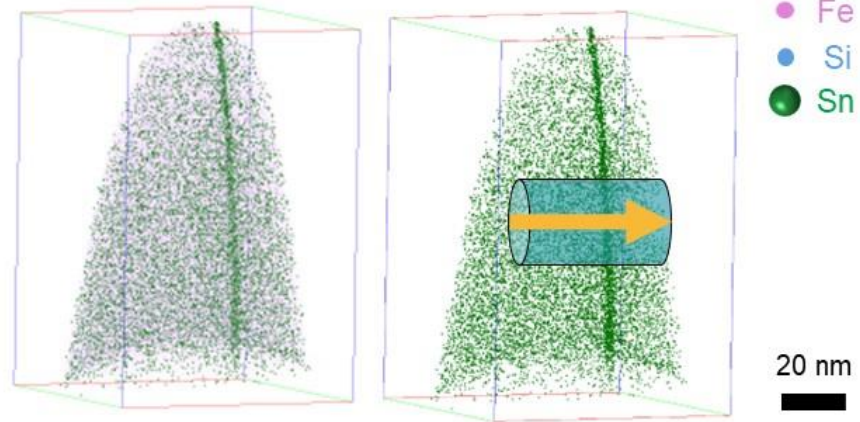
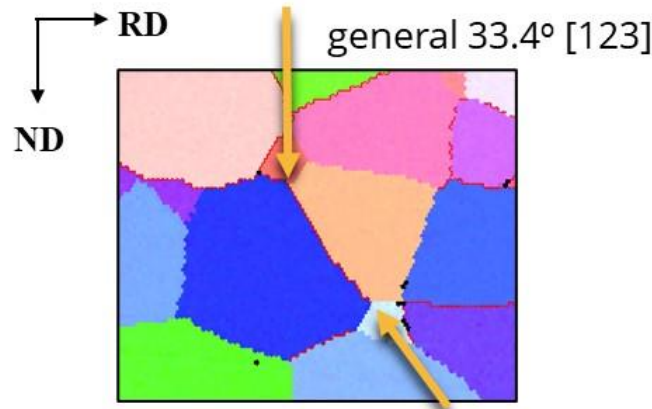
# Ségrégation de l'étain

## Méthodologie

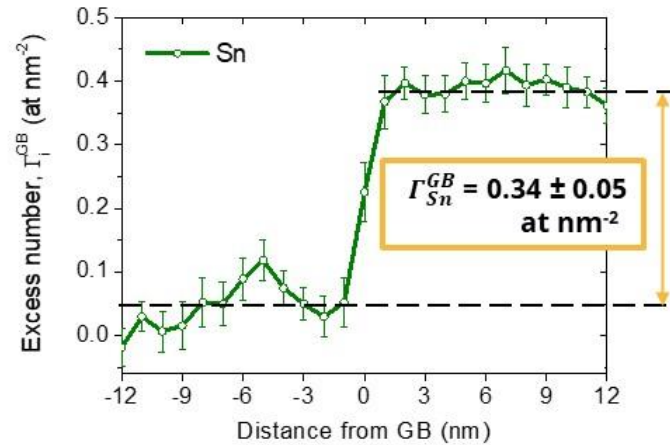
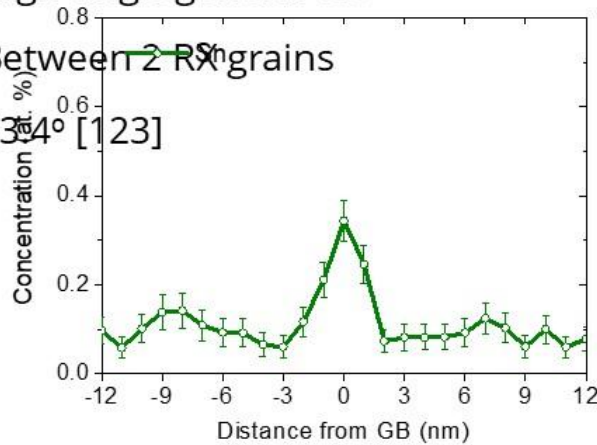


# Ségrégation de l'étain

## Ségrégation de Sn sur les joints de grains à l'état final



- High-angle general GB
- Between 2 RX grains
- $33.4^\circ [123]$

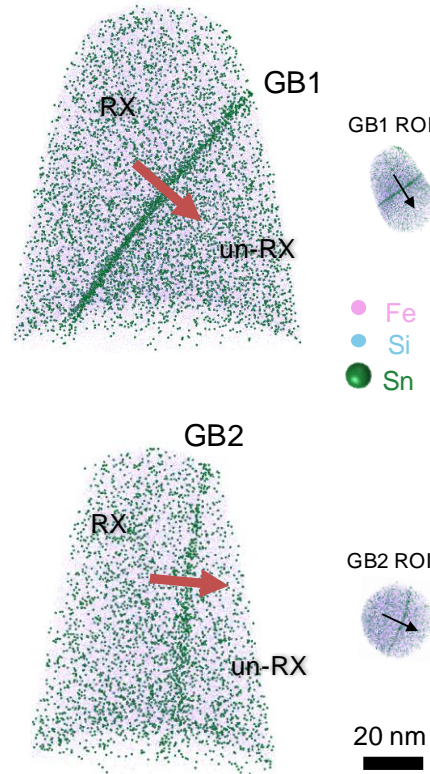
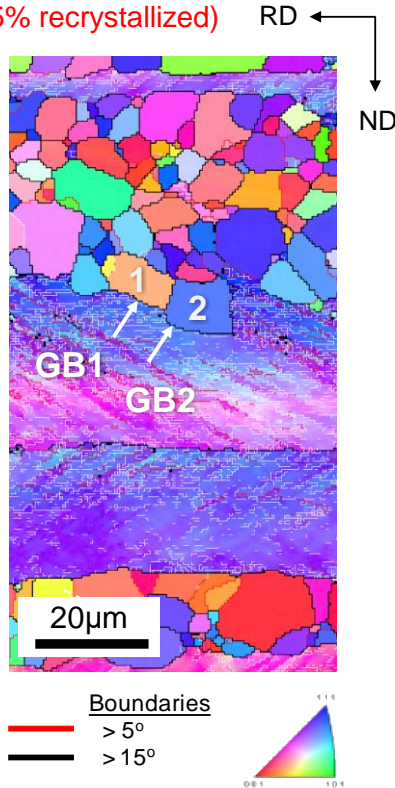


# Ségrégation de l'étain

## Observation de l'étain le long du front de recristallisation

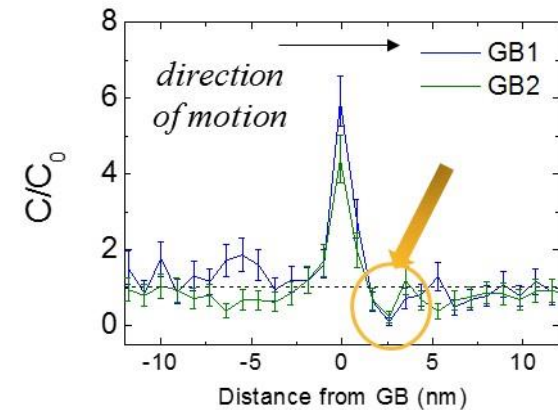
Solute drag and segregation

700°C – 10s  
(45% recrystallized)



Segregation at the recrystallization front:

	$\Gamma_{Sn}$ Excess (at/nm <sup>2</sup> )
GB1 $\theta = 48.2^\circ$	$0.60 \pm 0.03$
GB2 $\theta = 27.5^\circ$	$0.41 \pm 0.06$



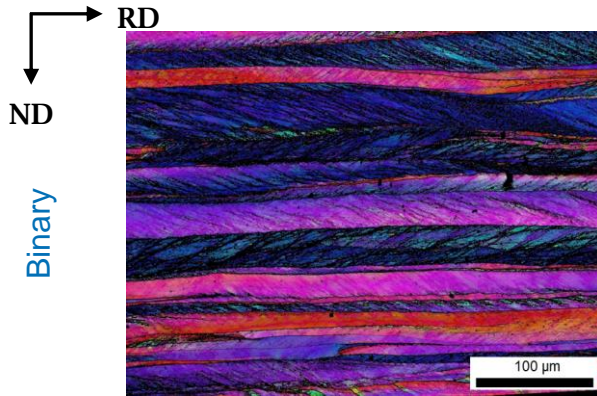
Depletion ahead of the recrystallization front

N. Mavrikakis et al. *Materialia*, Vol. 9, 2020, 100541.

# Effet sur la texture ?

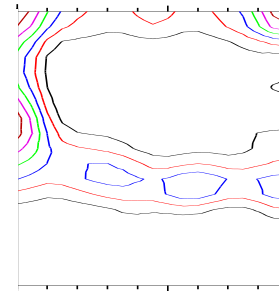
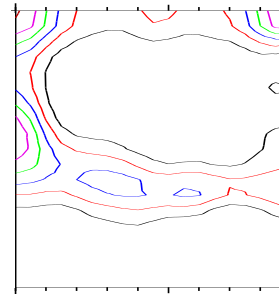
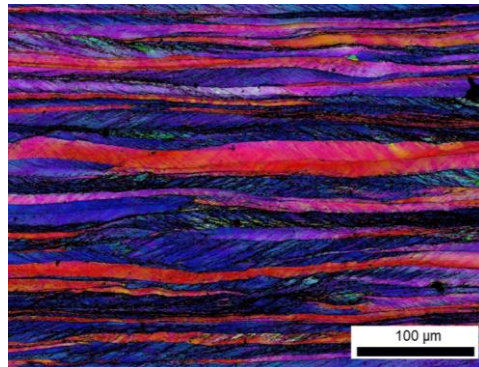
## Texture initiale dans les deux alliages

Initial state

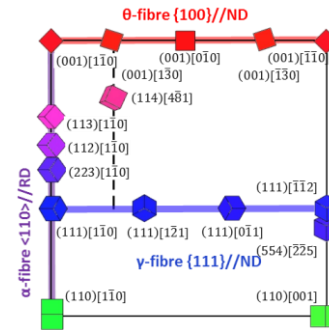


Binary

Ternary



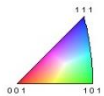
$\phi_2 = 45^\circ$



$\phi$

- 2.04
- 4.13
- 6.23
- 8.32
- 10.42
- 12.51

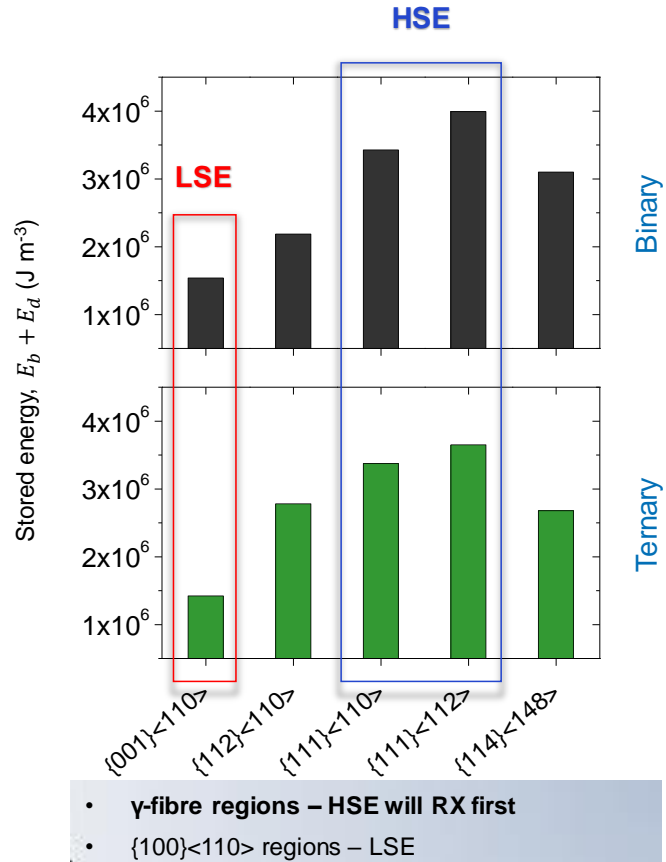
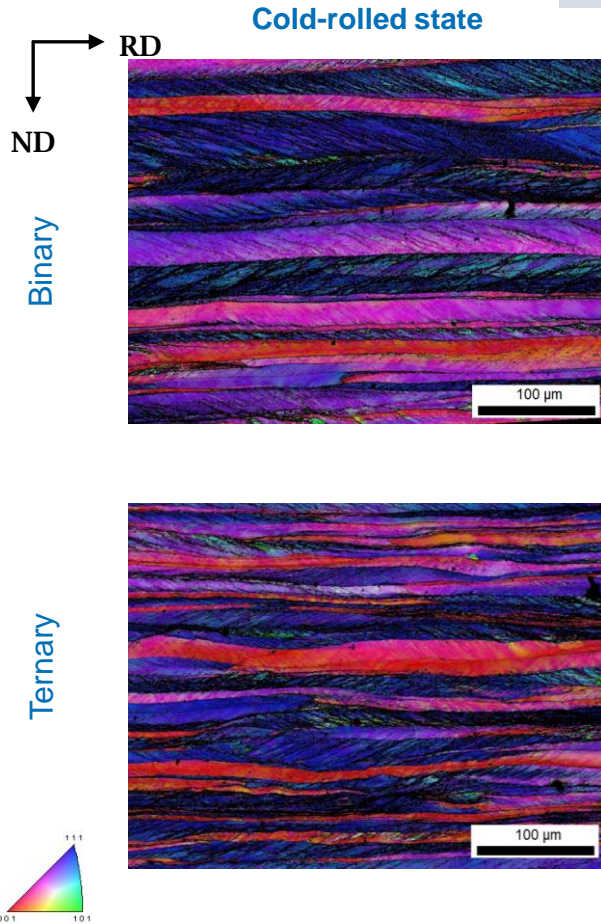
- Similar features
- Similar deformation textures



# Effet sur la texture ?

## Texture initiale dans les deux alliages

Initial texture

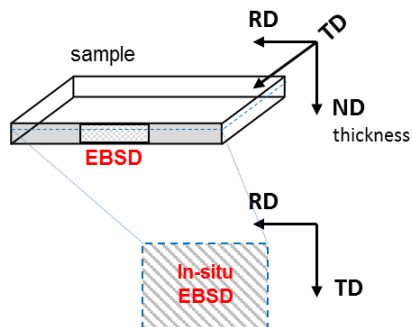


# Suivi in-situ de la recristallisation

## EBSD séquentiel

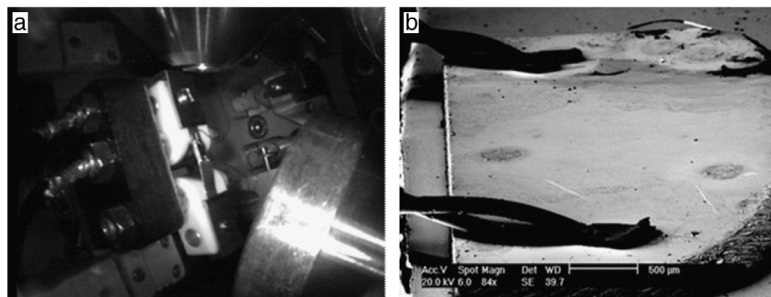
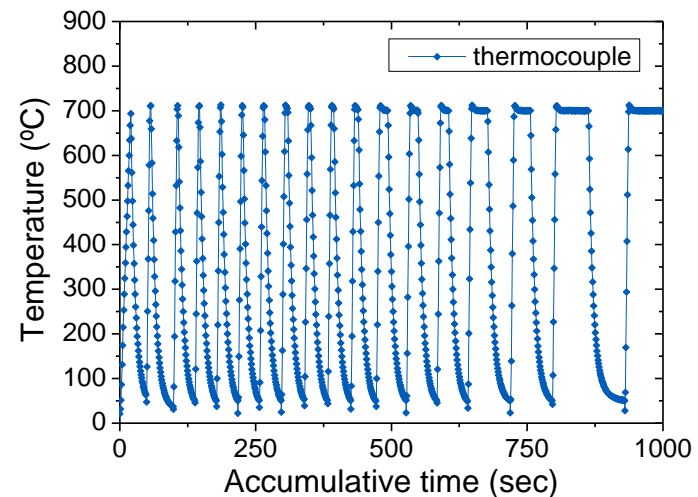
### Development of the recrystallization texture

- *in-situ* ReX of as-deformed materials
- Binary vs. Ternary



EBSD:  
900 x 300  $\mu\text{m}^2$

In-situ EBSD:  
400 x 600  $\mu\text{m}^2$



Experiments were done in collaboration with Prof. N. Bozzolo in MINES ParisTech, CEMEF, Sophia Antipolis

Heat-treatment inside SEM:

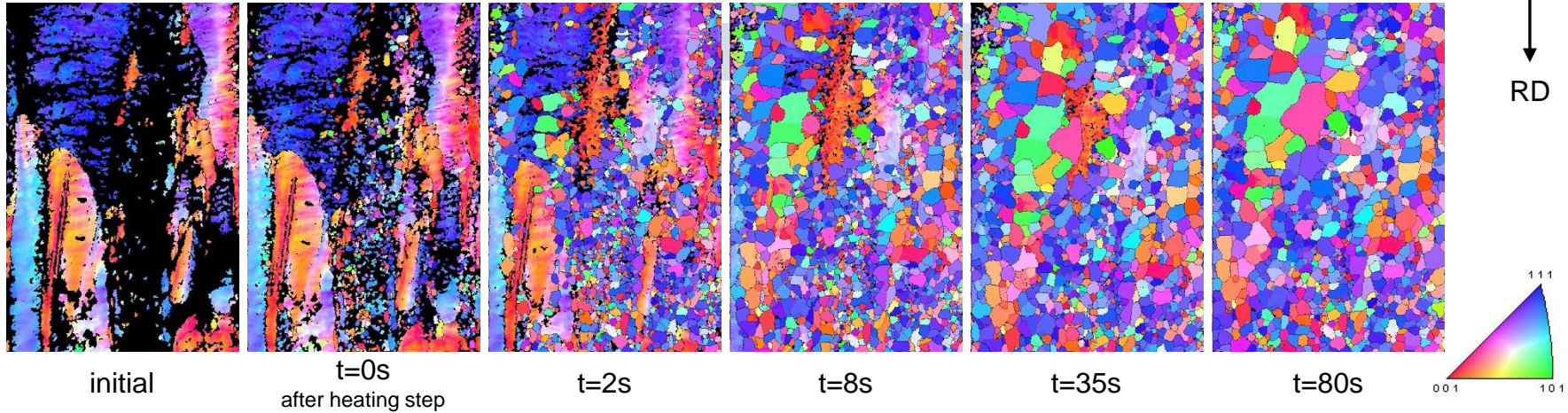
- 1<sup>st</sup> heating 25°C/s
- 2<sup>nd</sup>, 3<sup>rd</sup> ...n<sup>th</sup> heating ~ 100°C/s
- cooling faster than 80°C/s

# Suivi de la recristallisation

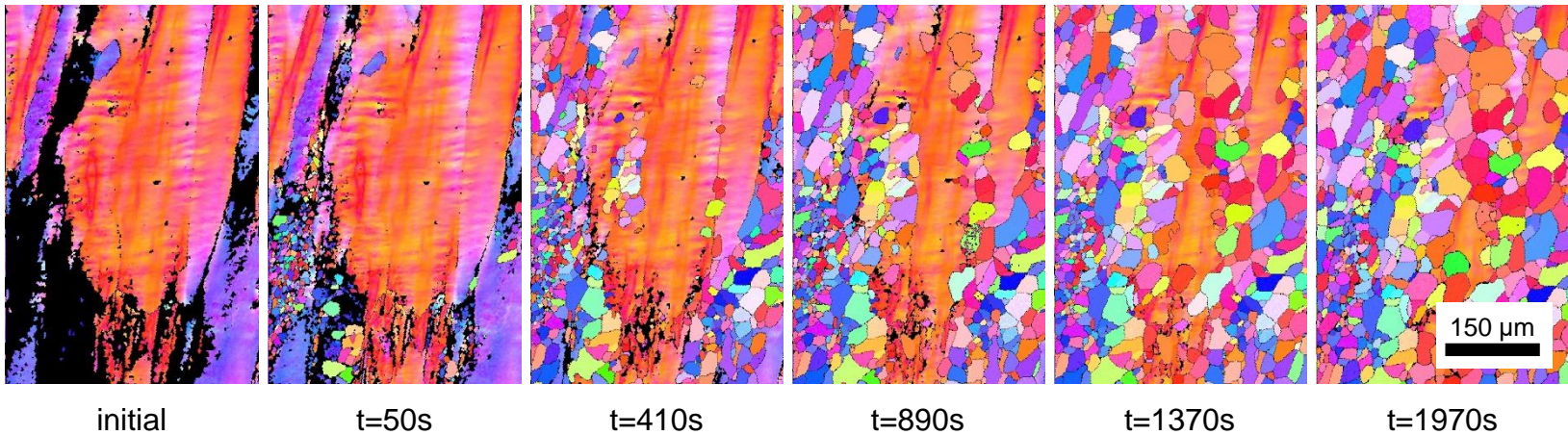
## EBSD séquentiel

Development of the recrystallization texture

Binary



Ternary



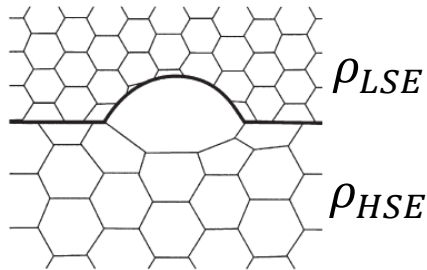
# Suivi de la recristallisation

## Mise en évidence d'un comportement différent

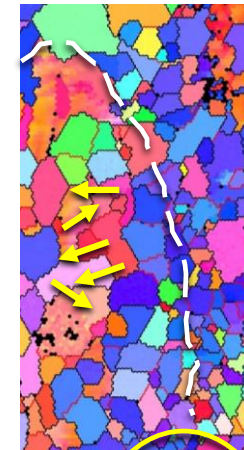
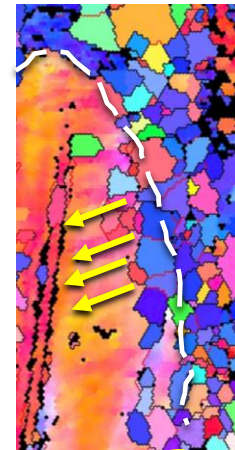
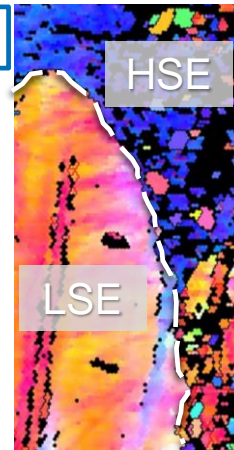
Development of the recrystallization texture

Effect on recrystallization texture

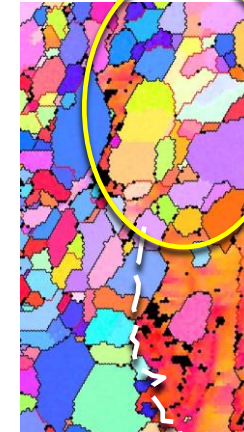
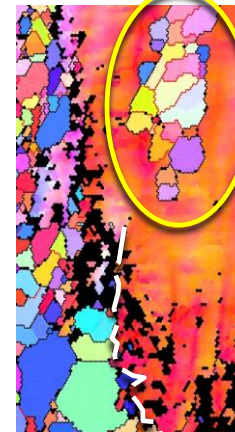
SIBM mechanism



Binary



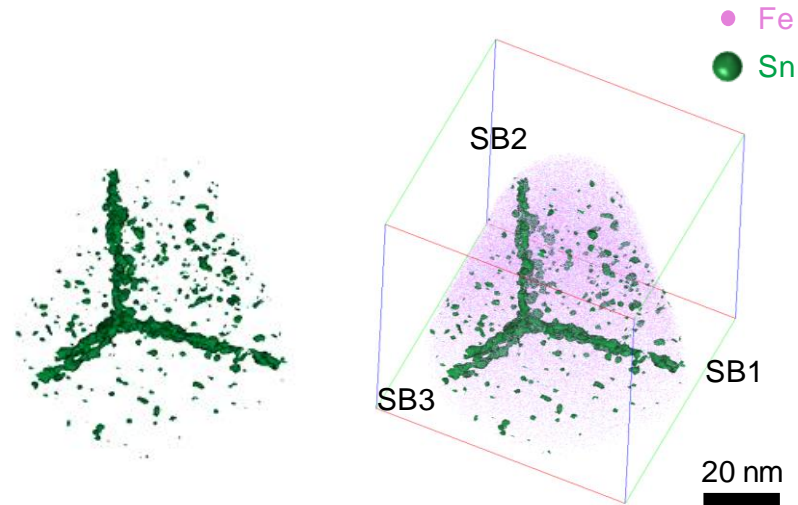
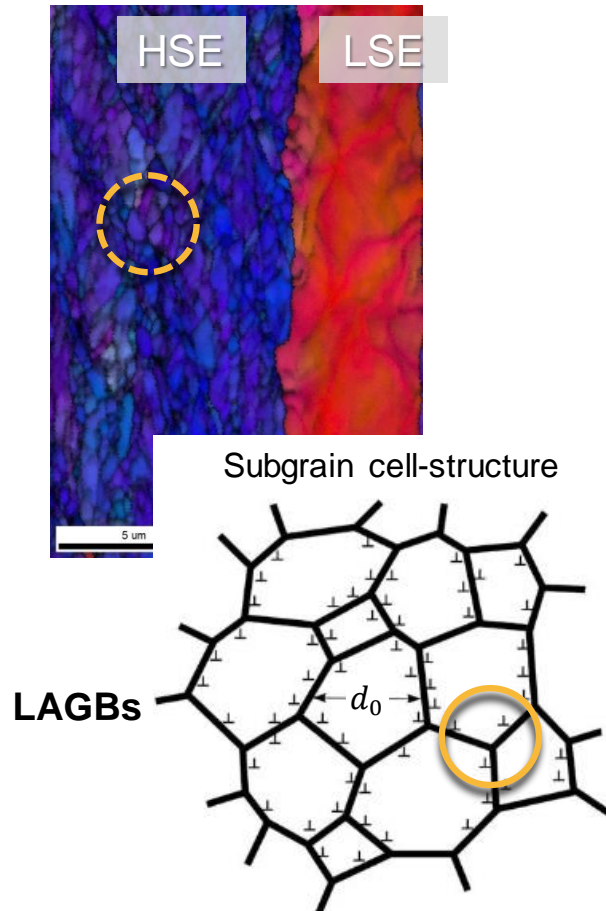
Ternary



- With Sn no SIBM observed
- LSE regions RX later

# Rôle de l'étain ?

## Ségrégation sur les sous-joints de grain dans les grains $\gamma$



Sn 0.5 at.-%-iso

Sn segregation at subgrain boundaries – consequences:

- Slow nucleation in HSE
- Later no SIBM into LSE

→ less  $\gamma$ -fibre grains in RX texture

# Lien ségrégation / recristallisation dans les alliages Fe-Si-Sn

## Conclusion

L'étude précédente a clairement montré:

- Le lien chimie / recristallisation dans les aciers Fe-Si-Sn
- L'importance de l'étude à différentes échelles
- L'effet de la segregation de l'étain sur les textures de recristallisation

