

Development of compositional-gradient metallic alloys for  
combinatorial investigation of microstructures

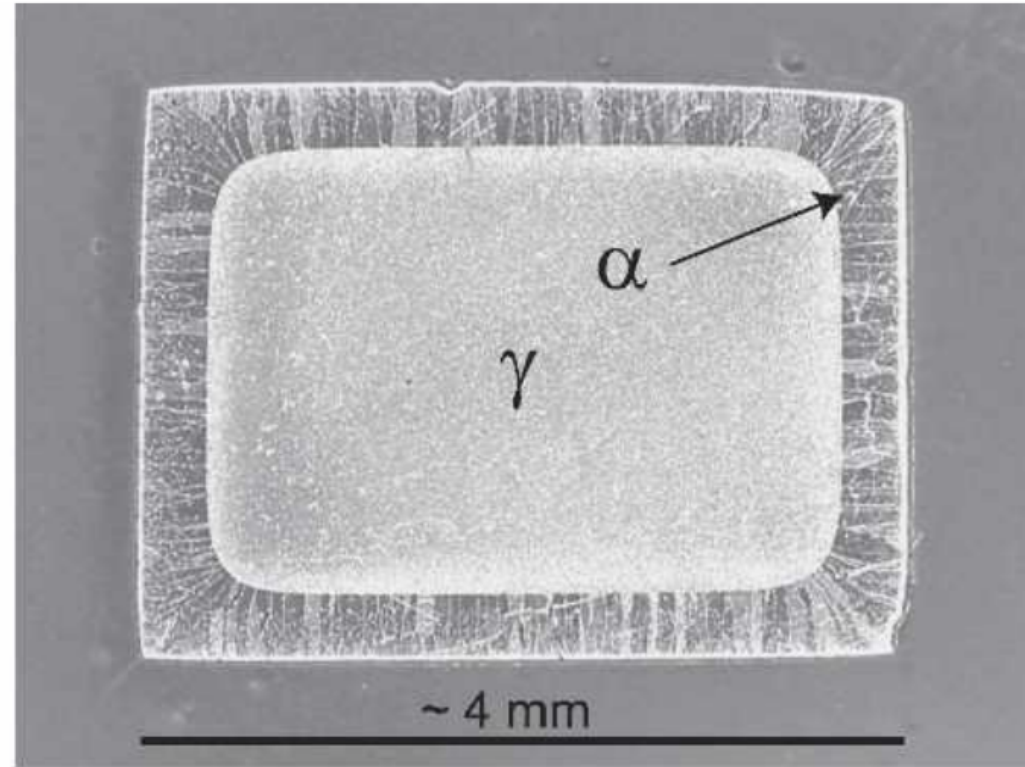
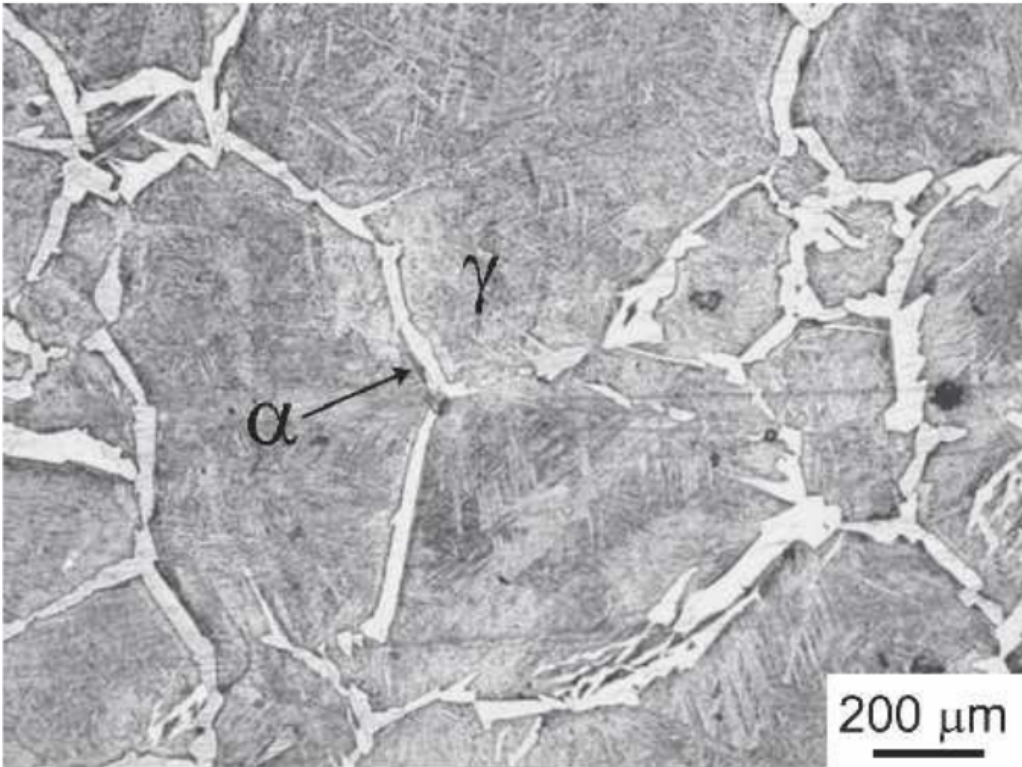
Imed-Eddine BENRABAH <sup>\*1</sup>, Hugo Paul VAN LANDEGHEM <sup>1</sup>, Alexis DESCHAMPS <sup>1</sup>, Frédéric BONNET <sup>2</sup>

<sup>1</sup> SIMAP, INP Grenoble, CNRS, St. Martin d'Hères Cedex, <sup>2</sup> Research, ArcelorMittal, Maizieres-les-Metz, France

ALEMI conference 13<sup>th</sup>-14<sup>th</sup> June, 2019, Vancouver, Canada

# Introduction - motivations

- Understanding and modeling of the influence of **composition** on ferrite growth kinetics during  $\gamma$  to  $\gamma + \alpha$  transformation.

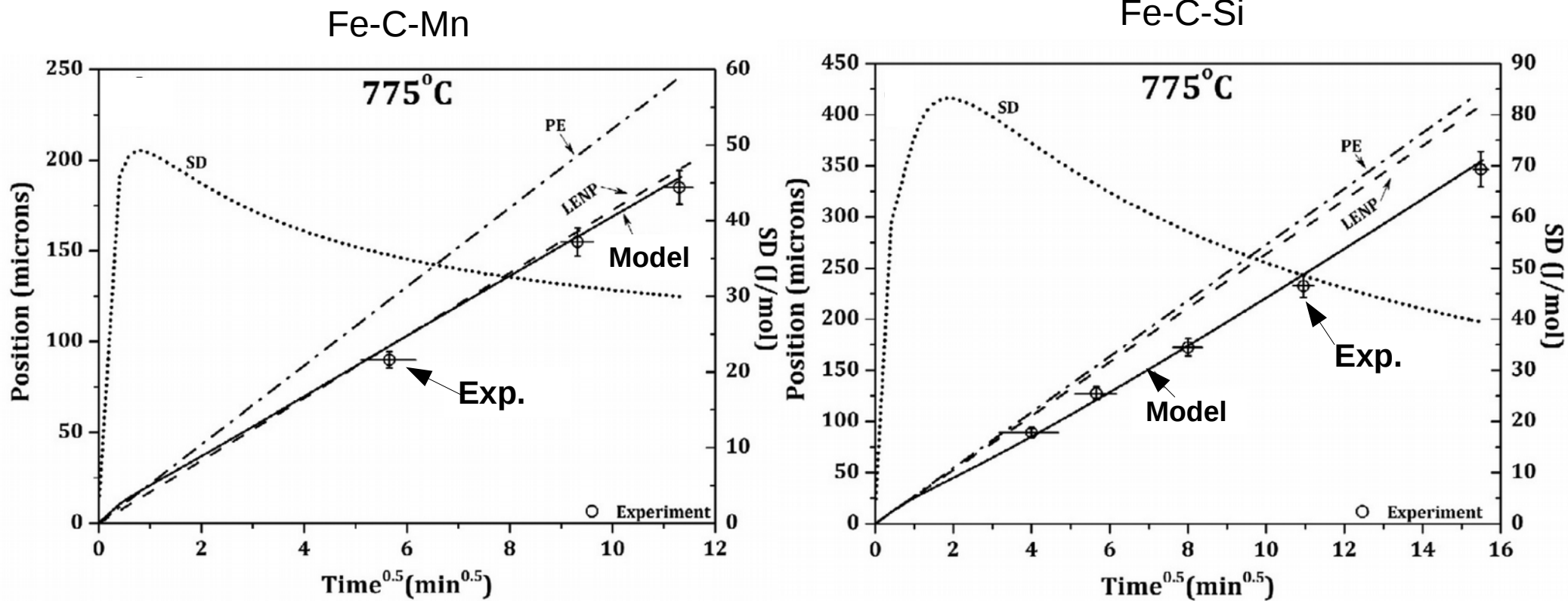


C.R. Hutchinson, H.S. Zurob, Y.J.M. Brechet, Metall. Mater. Trans. A 37A (2006) 1711–1720.

# Introduction-motivations

- Understanding and modeling of the kinetics of ferrite growth in multicomponent systems (Fe-C-X-Y-Z).

- Solute Drag based models for ternary systems



C Qiu, HS Zurob, CR Hutchinson - Acta Materialia, 2015

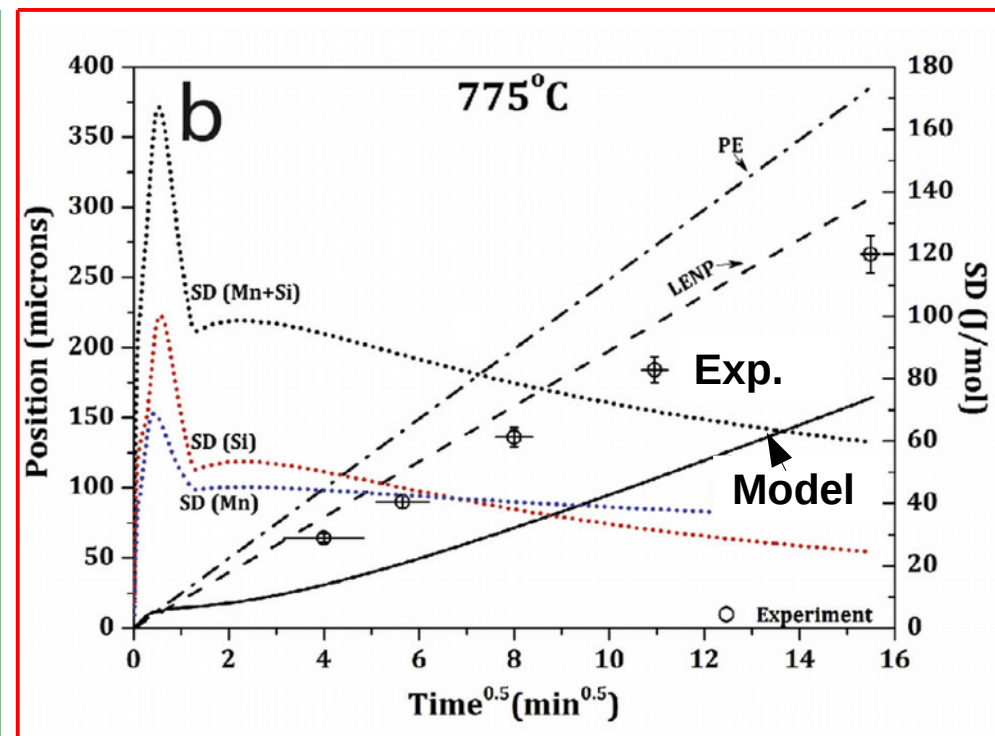
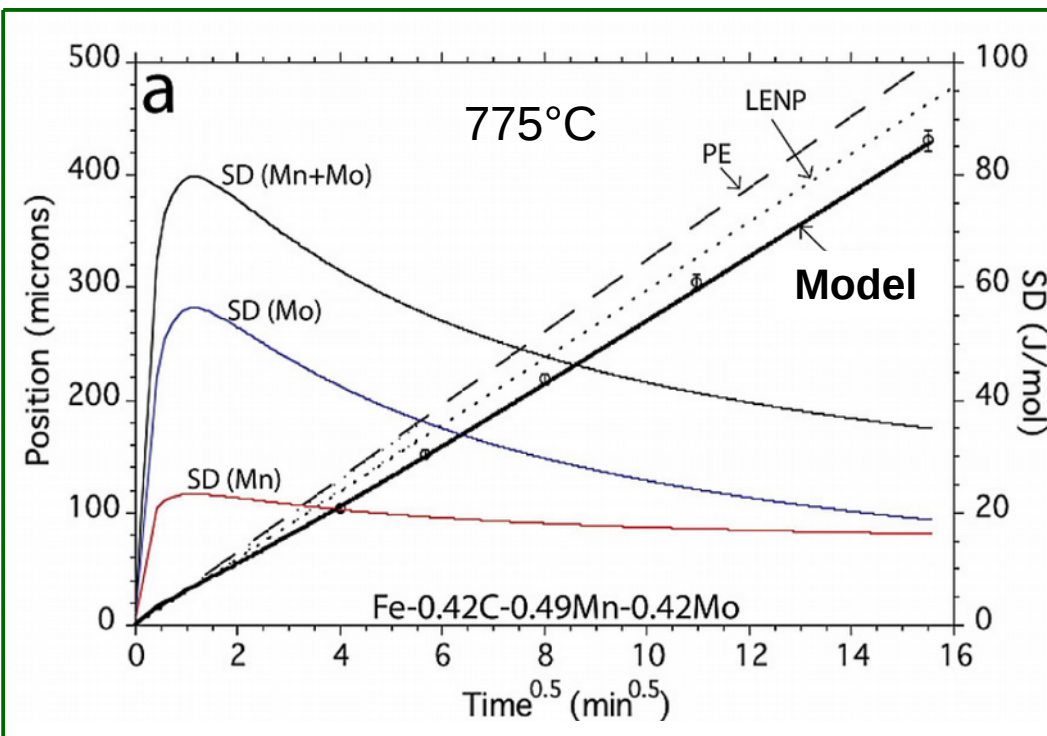
# Introduction-motivations

- Understanding and modeling of the kinetics of ferrite growth in multicomponent systems (Fe-C-X-Y-Z).

- Solute Drag based models for **Higher order systems**

Fe-C-Mn-Mo

Fe-C-Mn-Si



WW Sun, HS Zurob, CR Hutchinson - Acta Materialia, 2017

C Qiu, HS Zurob, CR Hutchinson - Acta Materialia, 2015

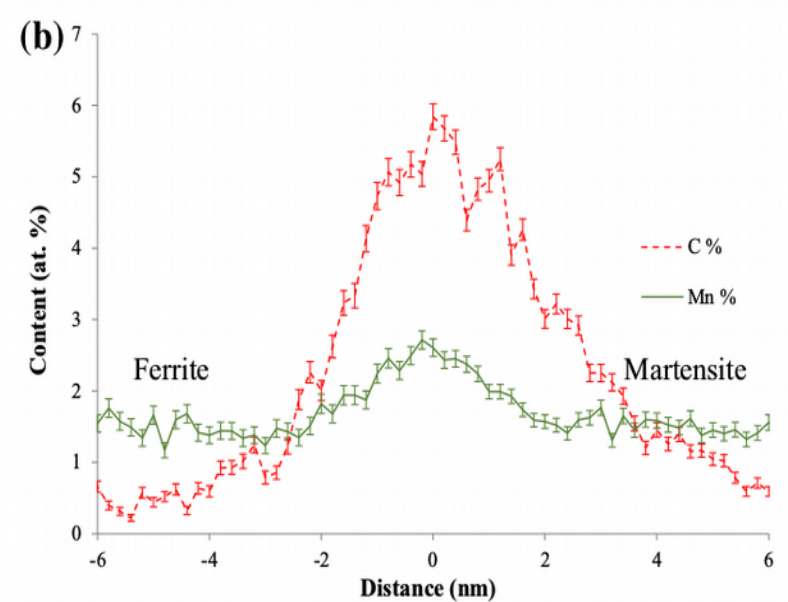
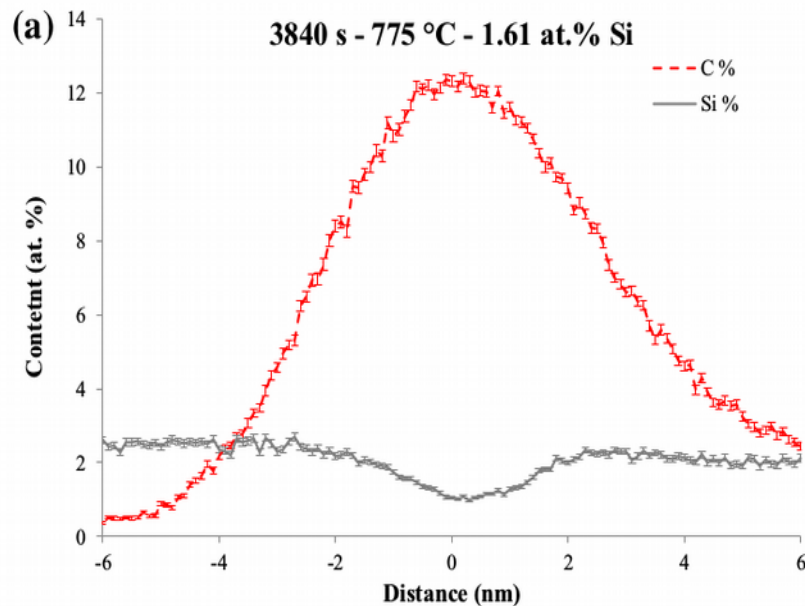
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- Understanding and modeling of the kinetics of ferrite growth in multicomponent systems (Fe-C-X-Y-Z).
- Solute segregation at the migrating interface (coupled or anti-coupled solute drag effect)

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- Understanding and modeling of the kinetics of ferrite growth in multicomponent systems (Fe-C-X-Y-Z).

- Solute segregation at the migrating interface (coupled or anti-coupled solute drag effect)
- Interaction between substitutional and interstitial atoms at the interface



Van Landeghem, H. P., Langelier, B., Panahi, D., Purdy, G. R., Hutchinson, C. R., Botton, G. A., & Zurob, H. S. (2016). JOM, 68(5).

# Introduction-motivations

- Implementing a new methodology for the fabrication of **compositionally-graded materials**.

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- Performing time- and space-resolved high energy X-ray diffraction experiments to gather phase transformation kinetics.



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In one composition gradient



Growth kinetics of **20** points of different compositions every **30** seconds



During **300** seconds  
**200** measurements per gradient



**Data for model calibration**

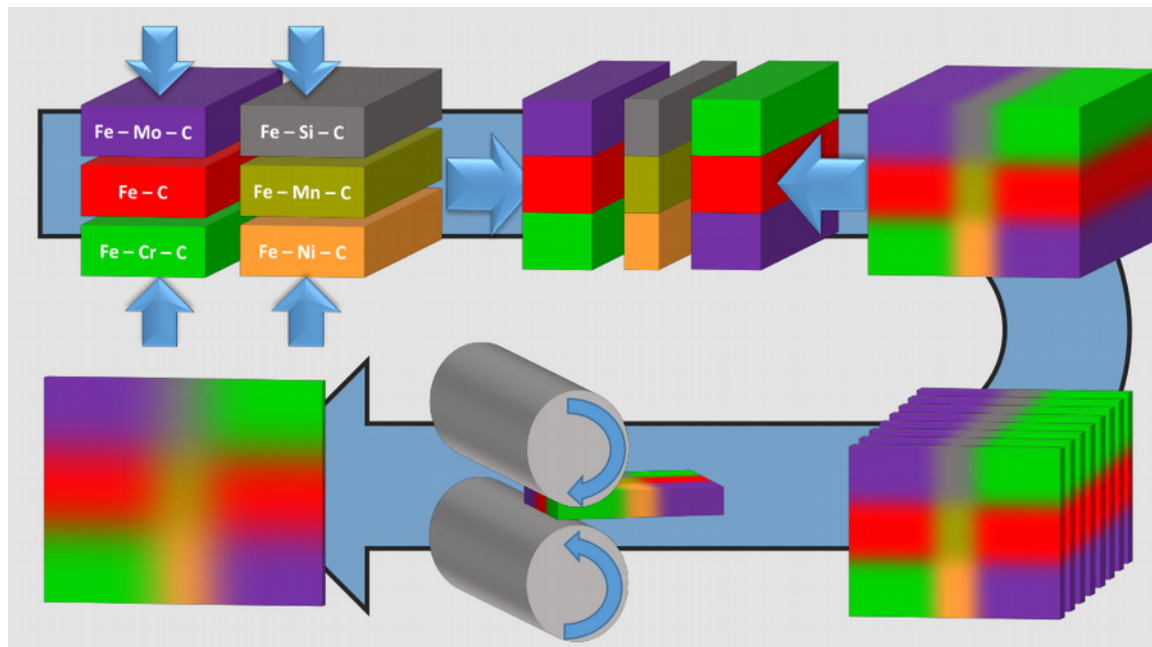
# Introduction-motivations

- Implementing a new methodology for the fabrication of **compositionally-graded materials**.
- Performing time- and space-resolved high energy X-ray diffraction experiments to gather phase transformation kinetics.
- APT (atom probe tomography) and TEM (transmission electron microscopy) techniques will be used on the treated samples to investigate the **segregation at the migrating interface**.

# Methodology of CGM fabrication

- Implementing a new methodology for the fabrication of **compositionally-graded materials**.

1 Solid joints  
Hot uniaxial compression



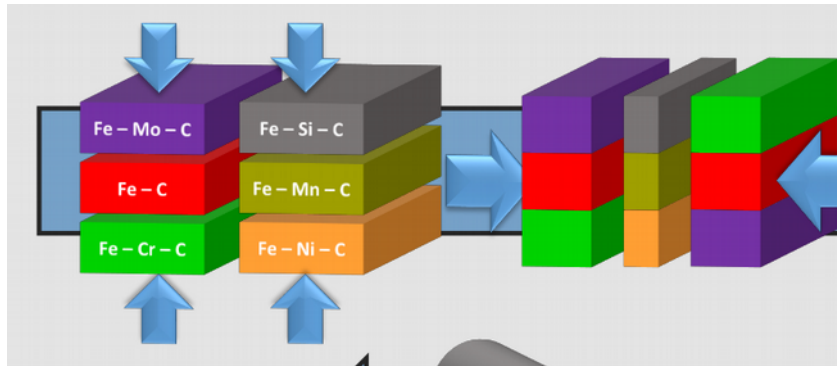
2 High temperature diffusion

3 Extending by cold/hot rolling

# Methodology of CGM fabrication

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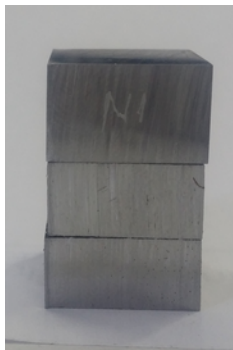
## 1 Solid joints Hot uniaxial compression



Fe-C-X1

Fe-C-X2

Fe-C-X3



Sample



Furnace

900°C  
22MPa  
1hour

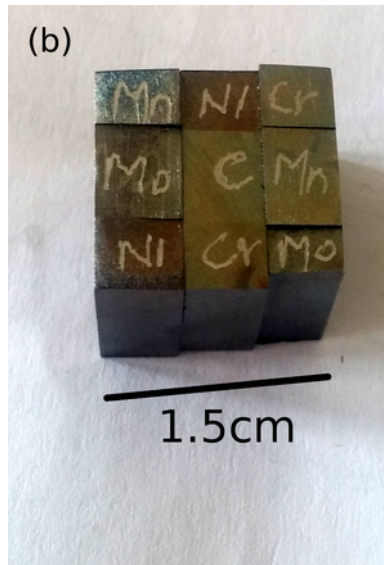
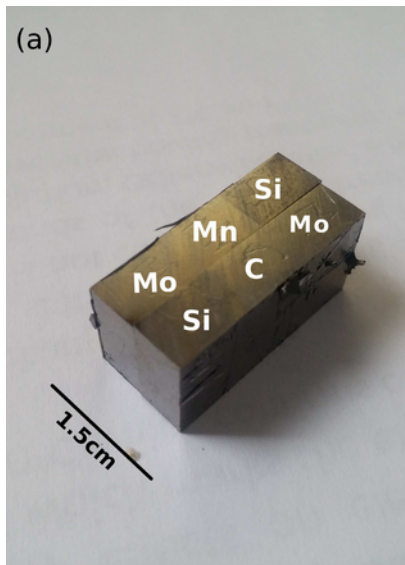
Applied  
weight



Ar/2%H<sub>2</sub>

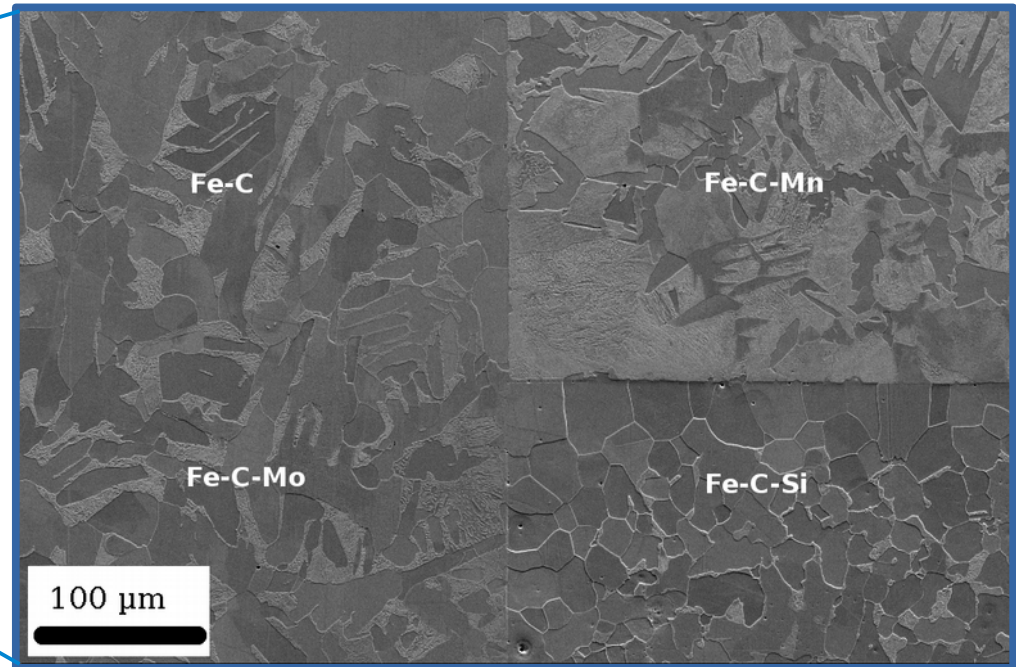
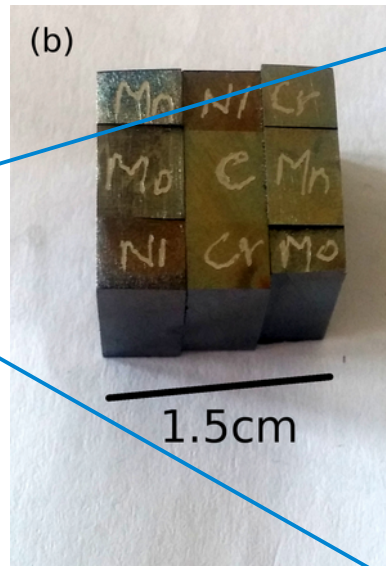
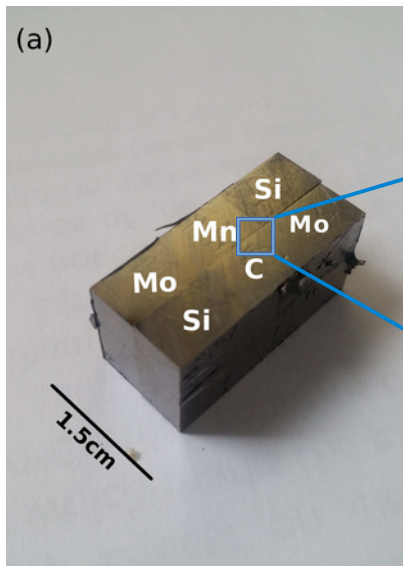
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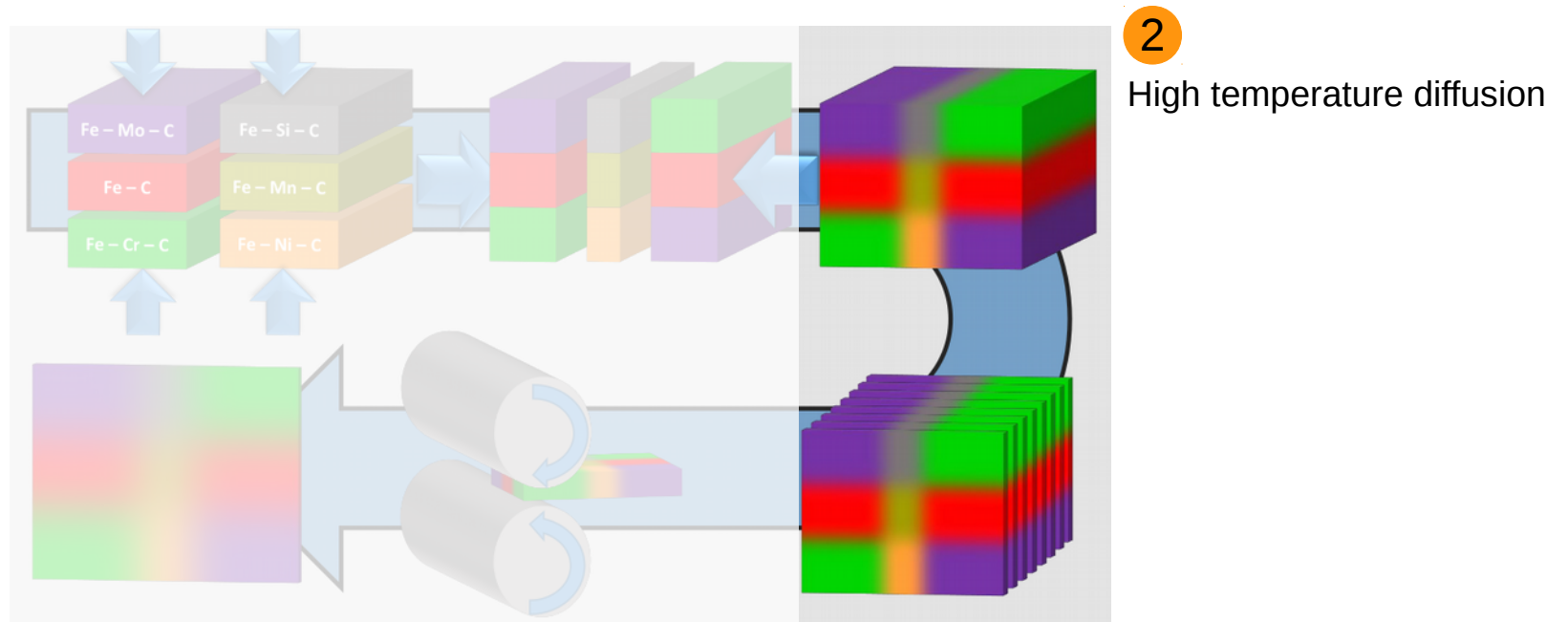
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# Methodology of CGM fabrication

- Implementing a new methodology for the fabrication of **compositionally-graded materials**.



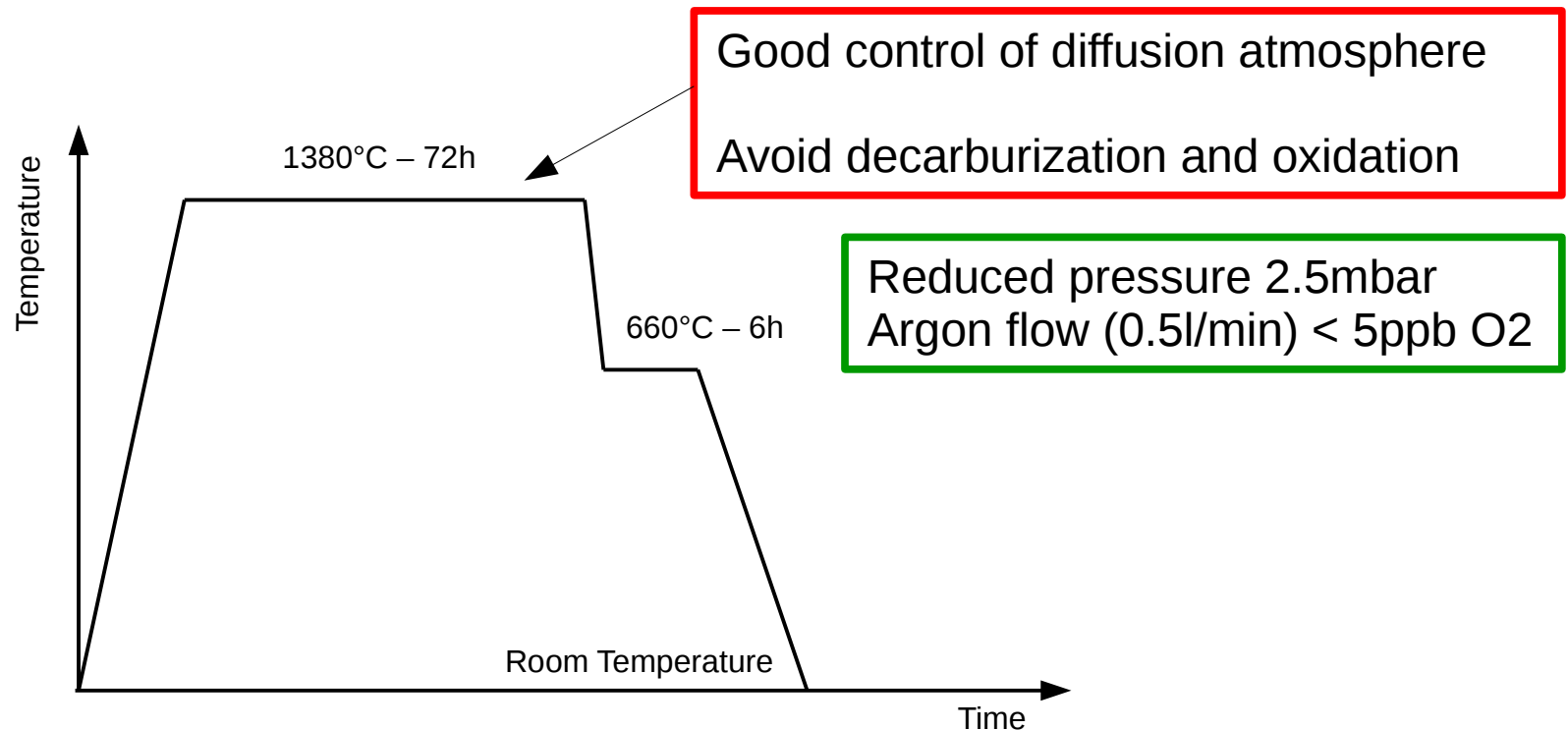
# Methodology of CGM fabrication

## 2 High temperature diffusion

Generate gradients of composition

Perform diffusion in the single phase domain

**DICTRA** 1380°C for 72 hours ~ 600-900 μm

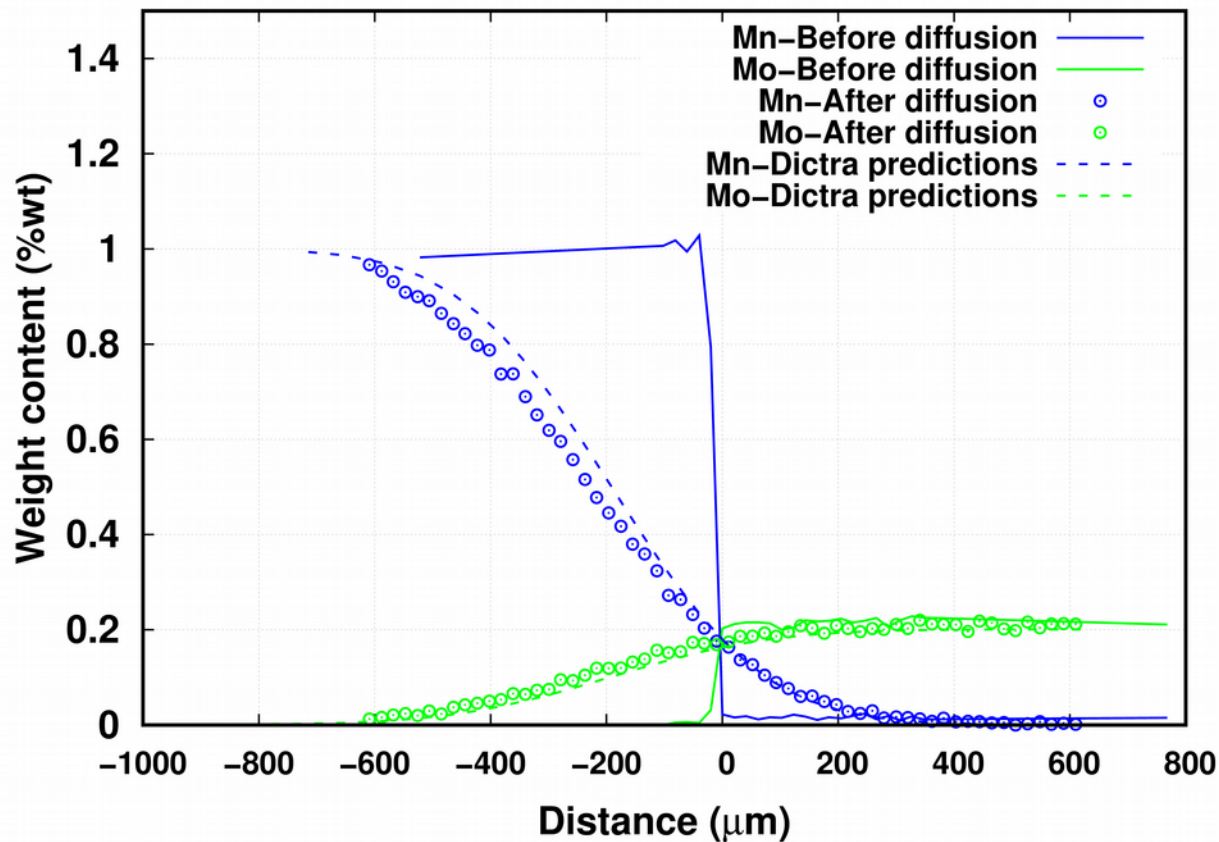
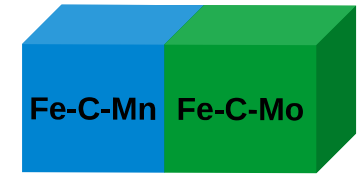




# Methodology of CGM fabrication

## 2 High temperature diffusion

EPMA measurements before and after diffusion treatment



Diffusion couple

Fe-0.3%C-1%Mn || Fe-0.3%C-0.2%Mo

72 hours at 1380°C

2.5mbar – Argon (high purity)

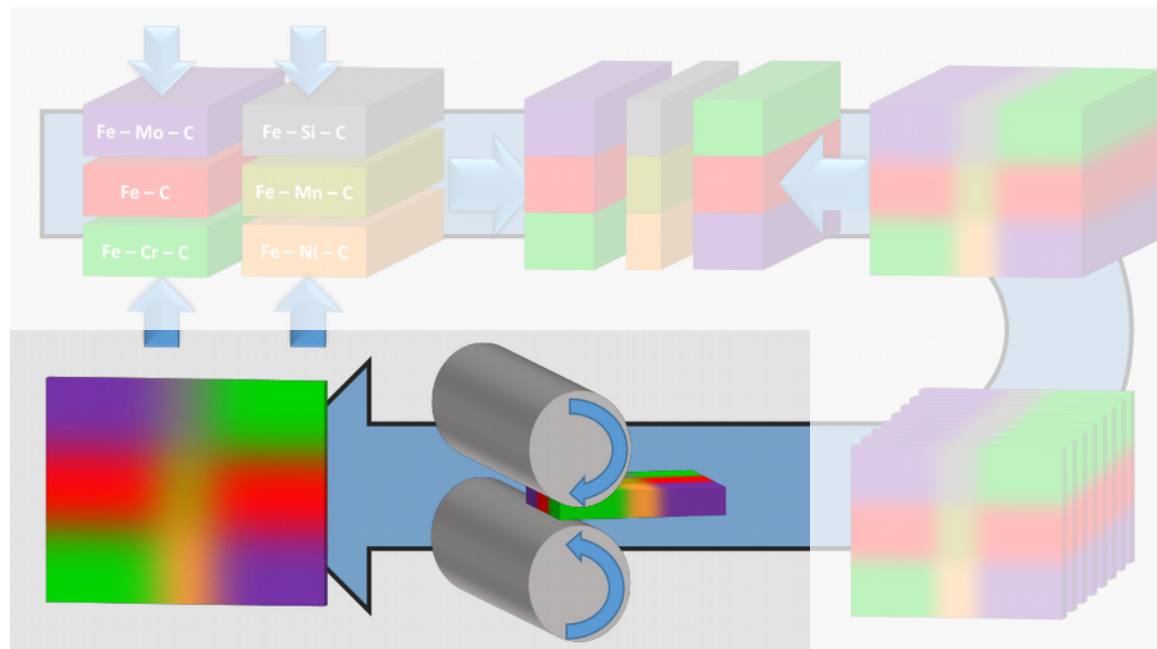
Profile length after diffusion

950μm (Mn profile)

700μm (Mo profile)

# Methodology of CGM fabrication

- Implementing a new methodology for the fabrication of **compositionally-graded materials**.

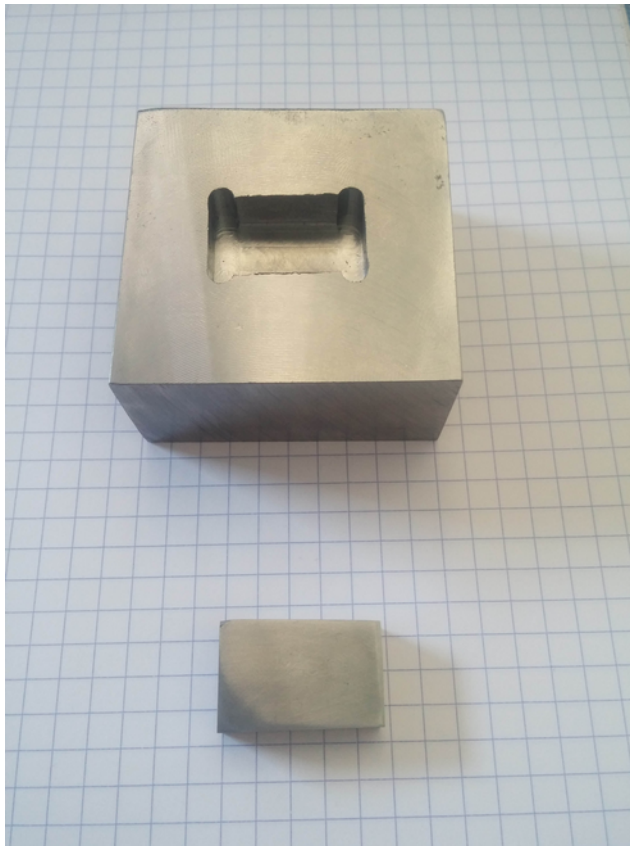


3 Extending by cold/hot rolling

# Methodology of CGM fabrication

## 3 Extending by cold/hot rolling

Low carbon steel



Sample

Reduction ~85%

Thickness : 7mm – to – 1mm

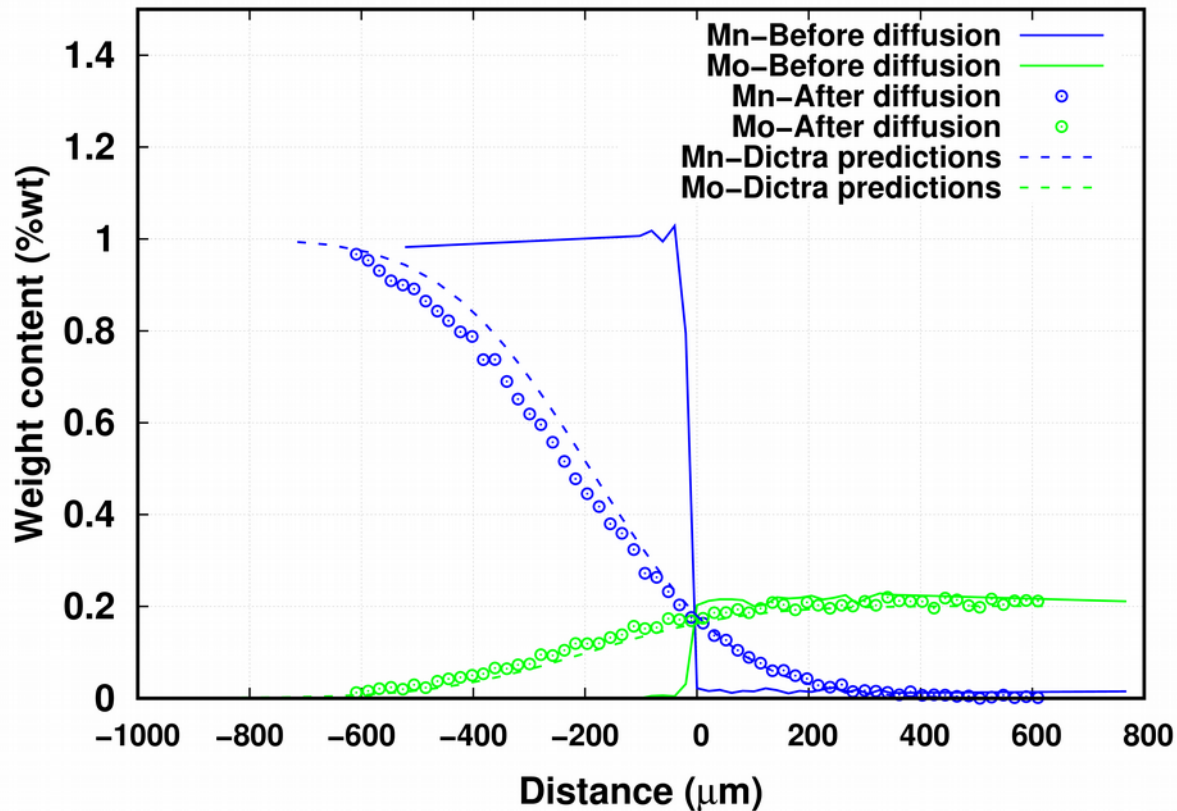
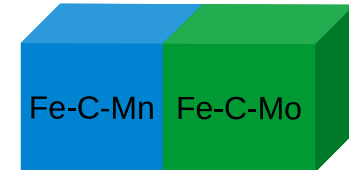


Small multiple cold rolling passes

# Methodology of CGM fabrication

## 3 Extending by cold/hot rolling

EPMA measurements before and after diffusion treatment



Diffusion couple

Fe-0.3%C-1%Mn || Fe-0.3%C-0.2%Mo

Profile length after diffusion

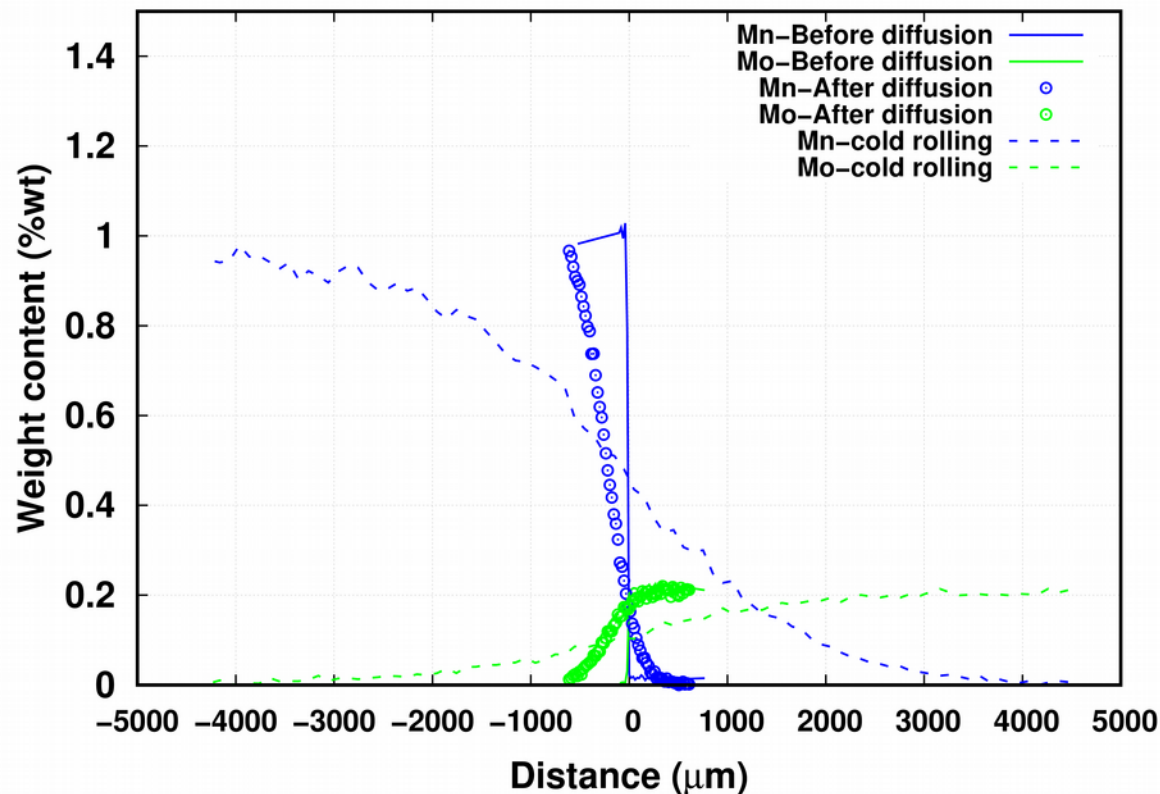
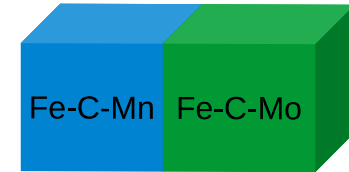
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EPMA measurements before and after diffusion treatment



Diffusion couple

Fe-0.3%C-1%Mn || Fe-0.3%C-0.2%Mo

Profile length after diffusion

950 $\mu\text{m}$  (Mn profile)

700 $\mu\text{m}$  (Mo profile)

After cold rolling

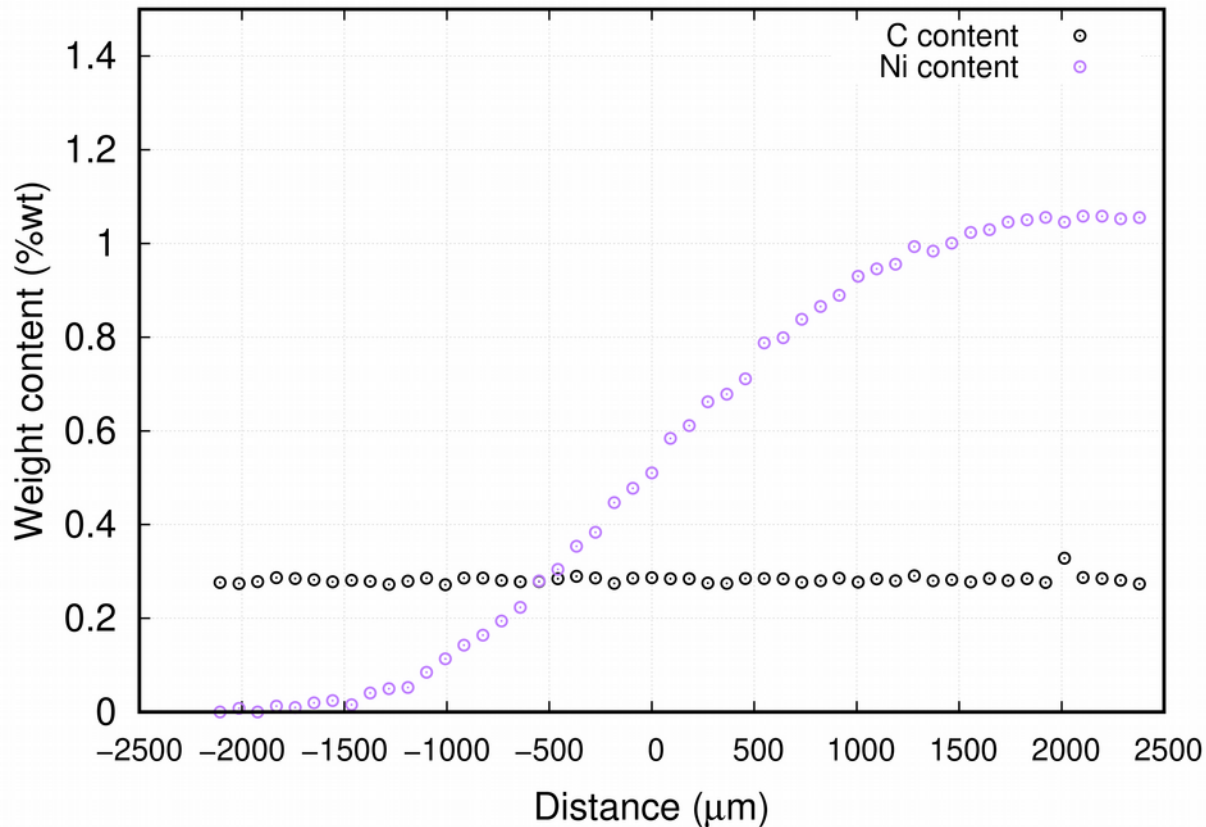
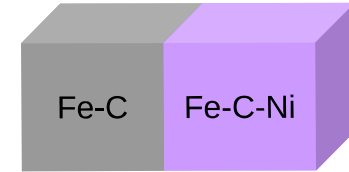
~7 mm (Mn profile)

~4.5mm (Mo profile)

# Methodology of CGM fabrication

## 3 Extending by cold/hot rolling

EPMA measurements after cold rolling



Diffusion couple

Fe-0.3%C || Fe-0.3%C-1.0%Ni

After cold rolling  
~3 mm (Ni profile)

# High energy X-ray diffraction experiments

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- Implementing a new methodology for the fabrication of **compositionally-graded materials**.
- Performing time- and space-resolved high energy X-ray diffraction experiments to gather phase transformation kinetics.

Synchrotron beam time : 12 shifts – 9<sup>th</sup>-12<sup>th</sup> November 2018

ESRF ( beamline ID11, HEXRD 87keV, beam size 200-600 $\mu$ m)





# High energy X-ray diffraction experiments

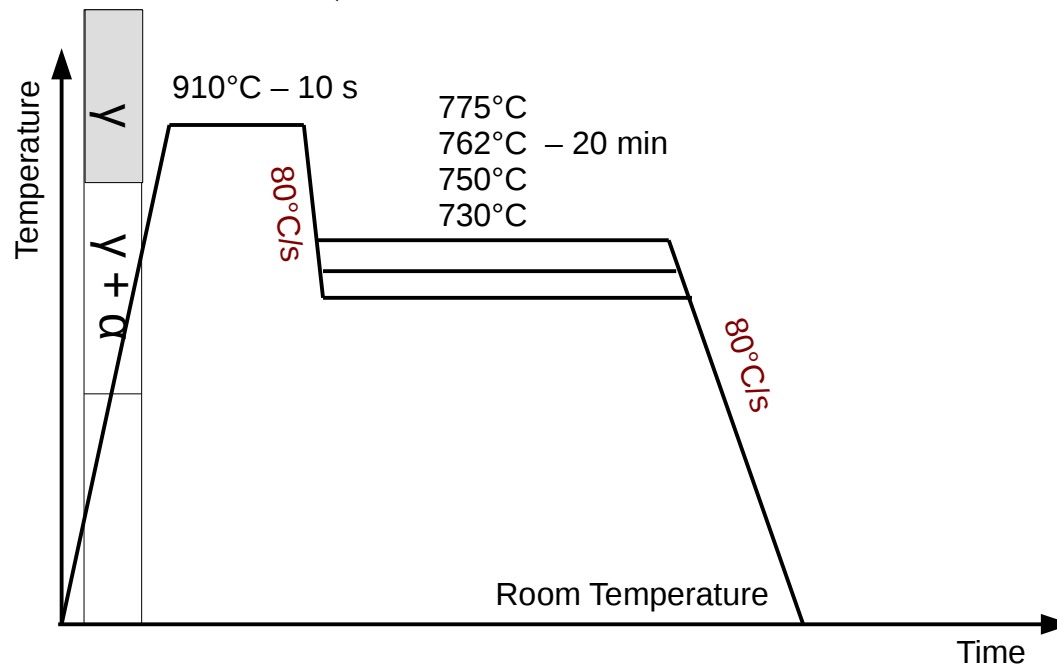
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Procedure :



# High energy X-ray diffraction experiments

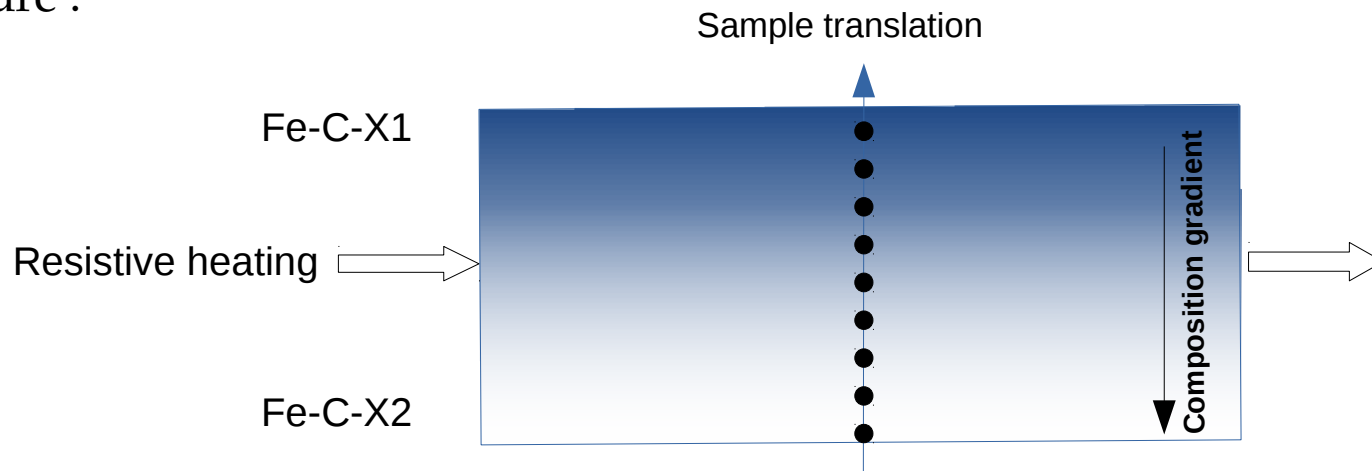
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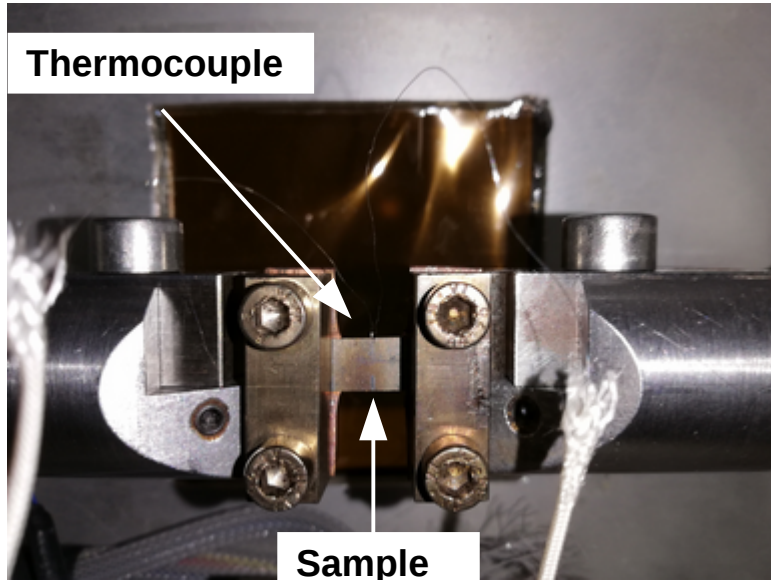
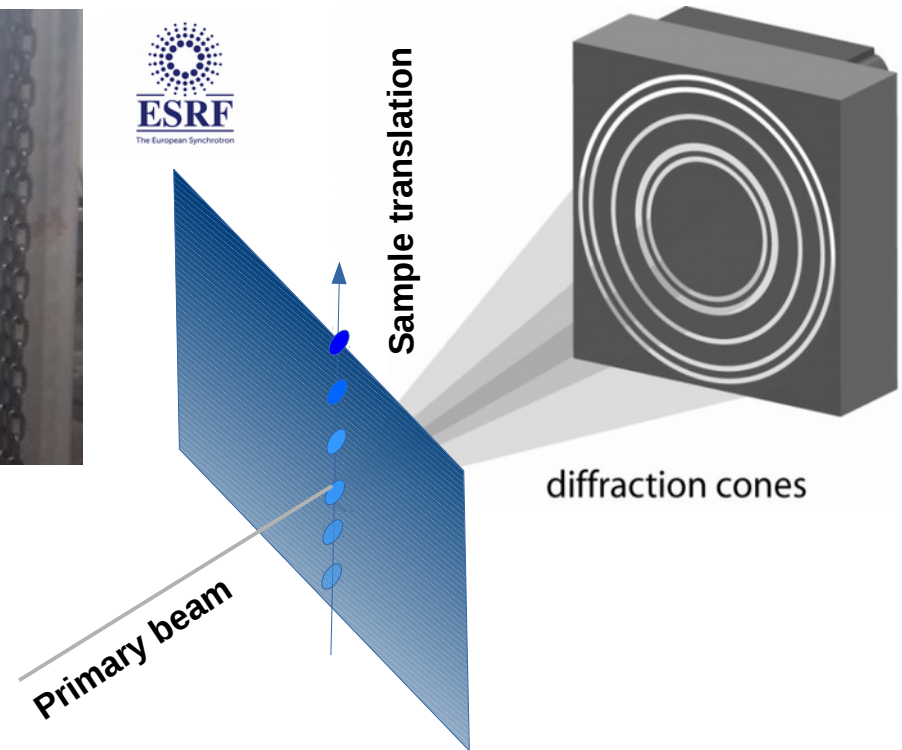
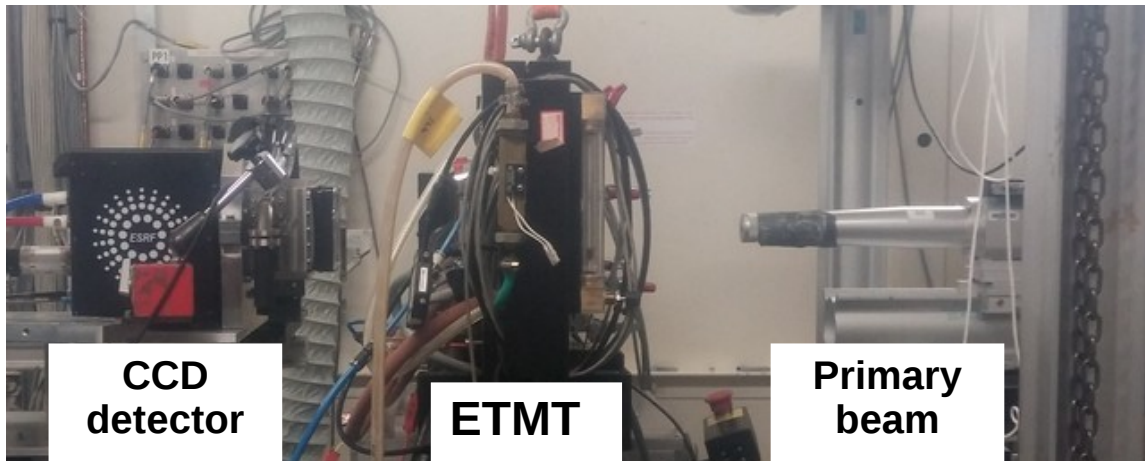
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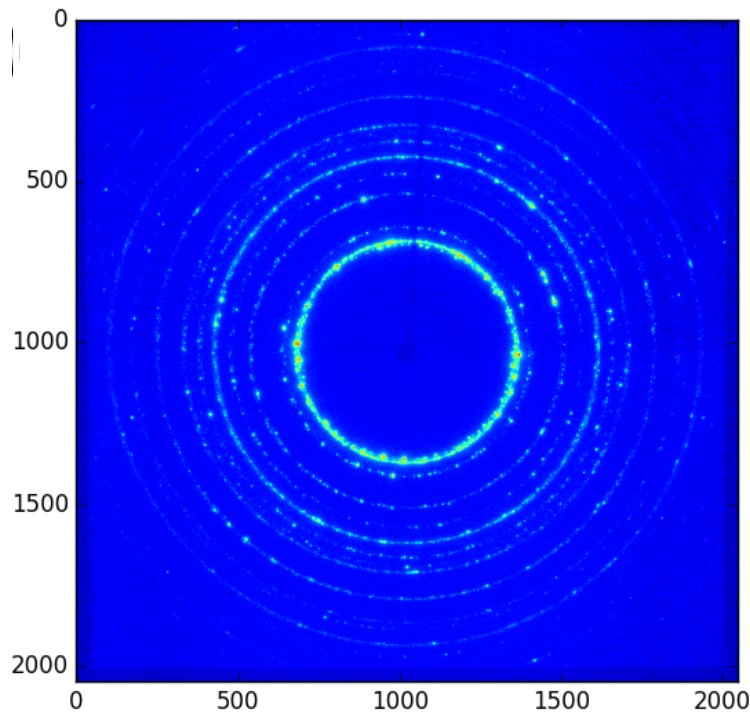


# High energy X-ray diffraction experiments

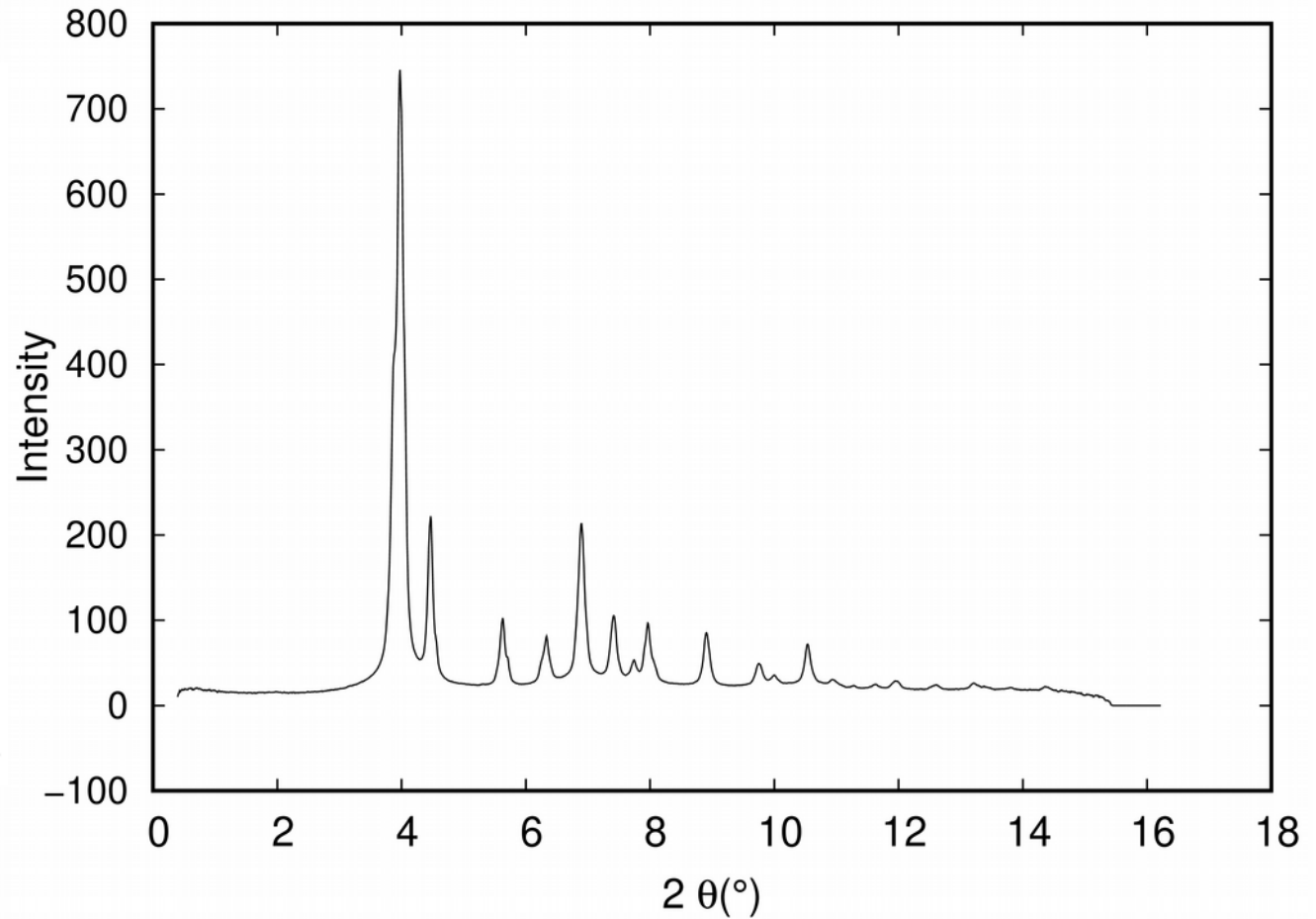


- Resistive heating
- 2D detector
- 2 welded thermocouples
- Controlled atmosphere : Argon
- Acquisition time : 0.15 s
- Scan over 3 to 8 mm
- Scan speed : 1mm / 2.6 s
- Scan step : 75  $\mu$ m

# High energy X-ray diffraction experiments



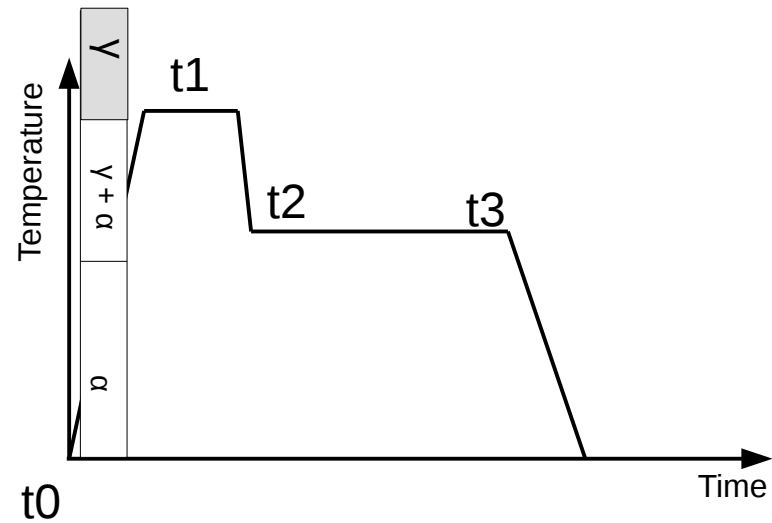
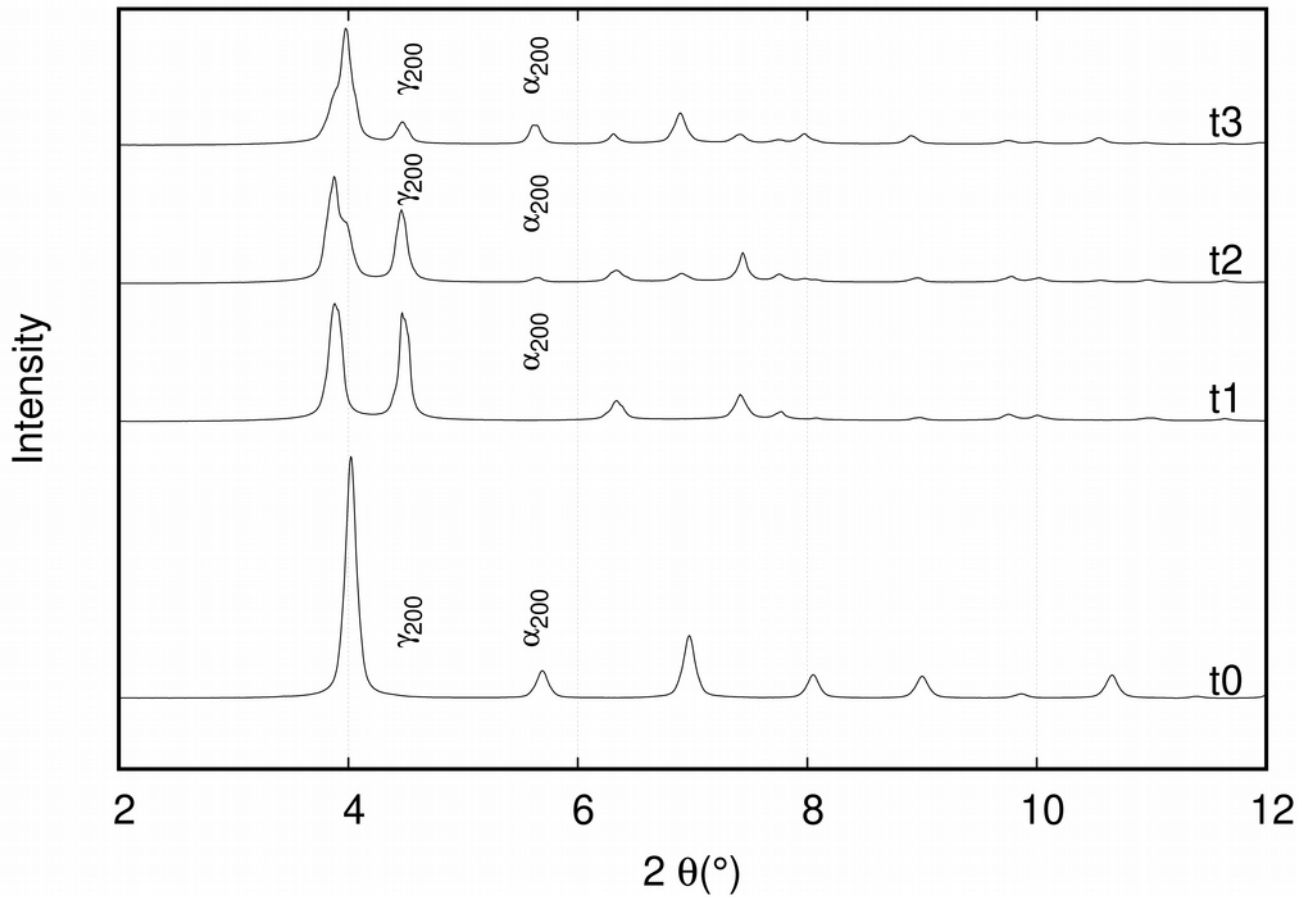
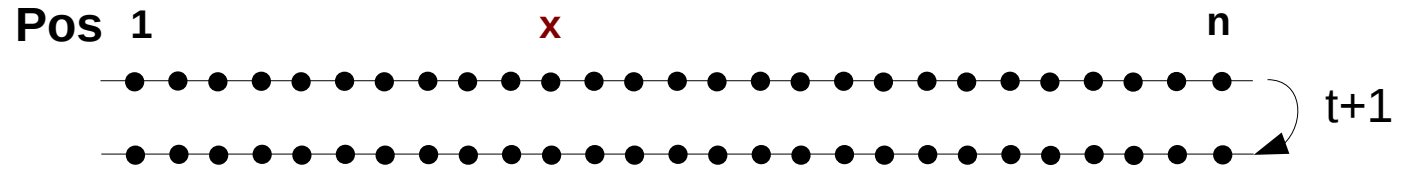
Debye-Scherrer diffraction rings



Intensity –  $2\theta$  plot  
circular integration

# Results

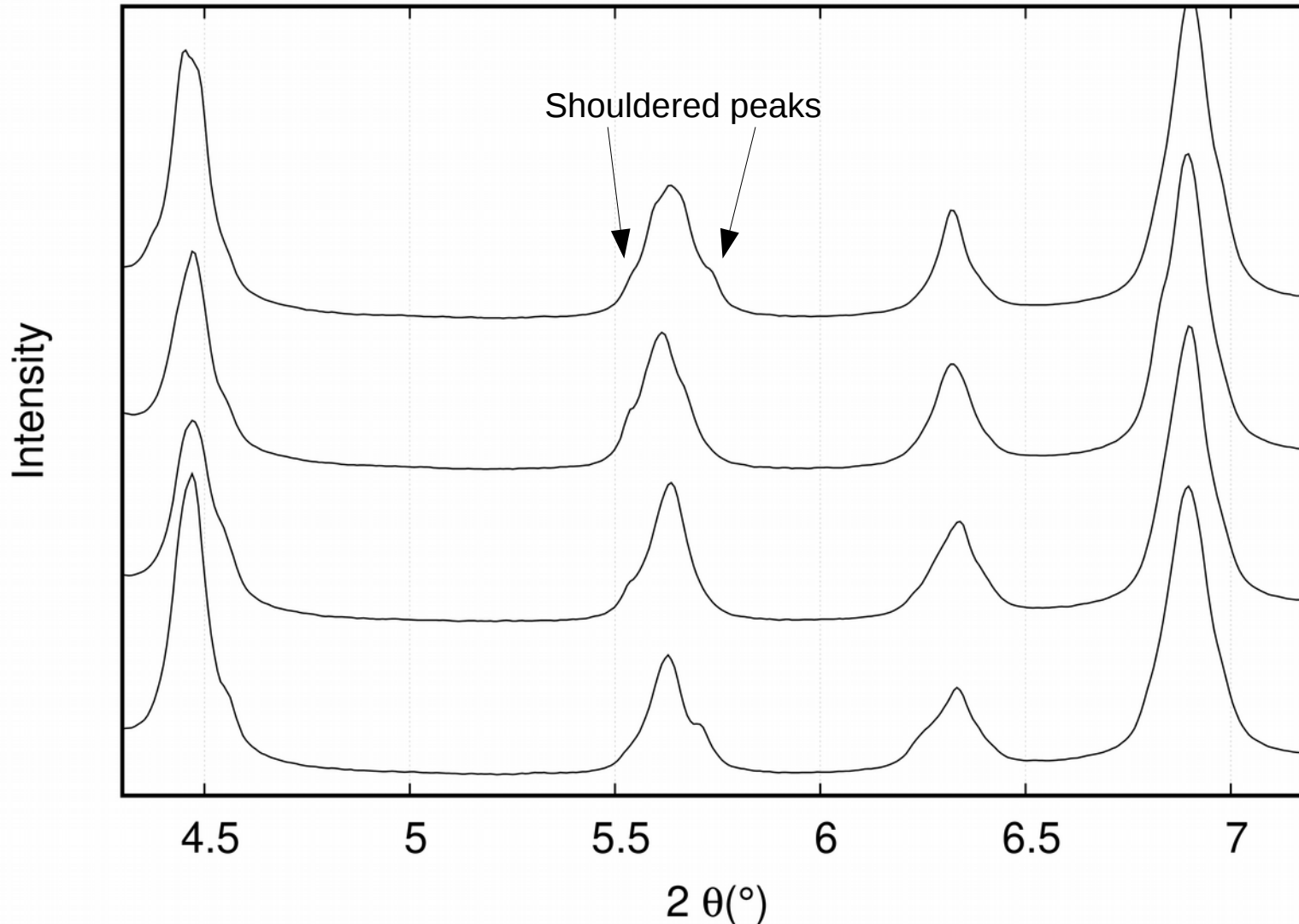
Data analysis :



# Results

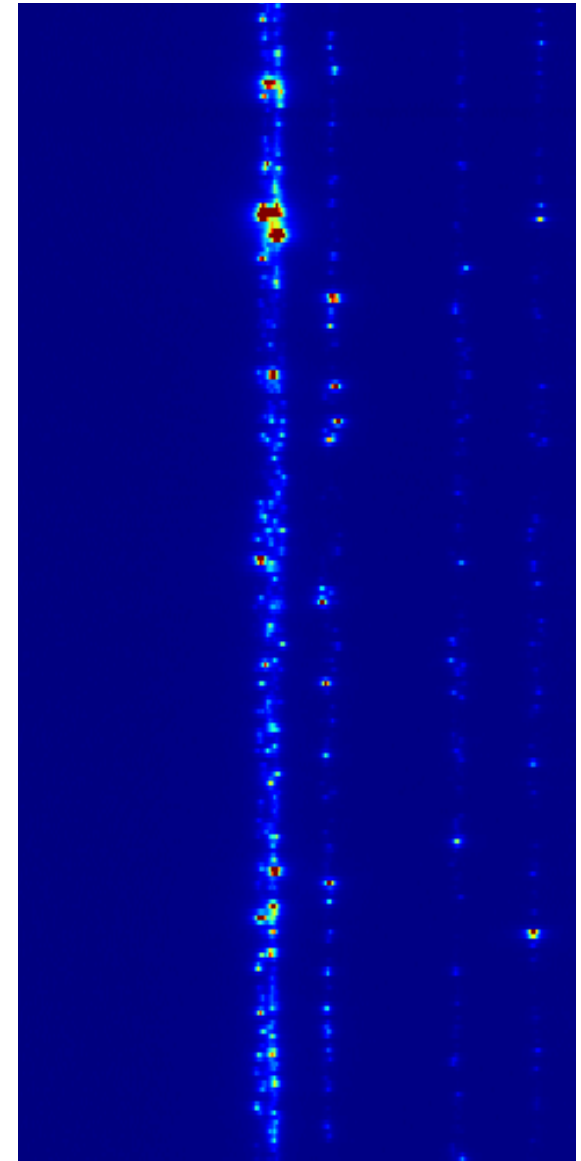
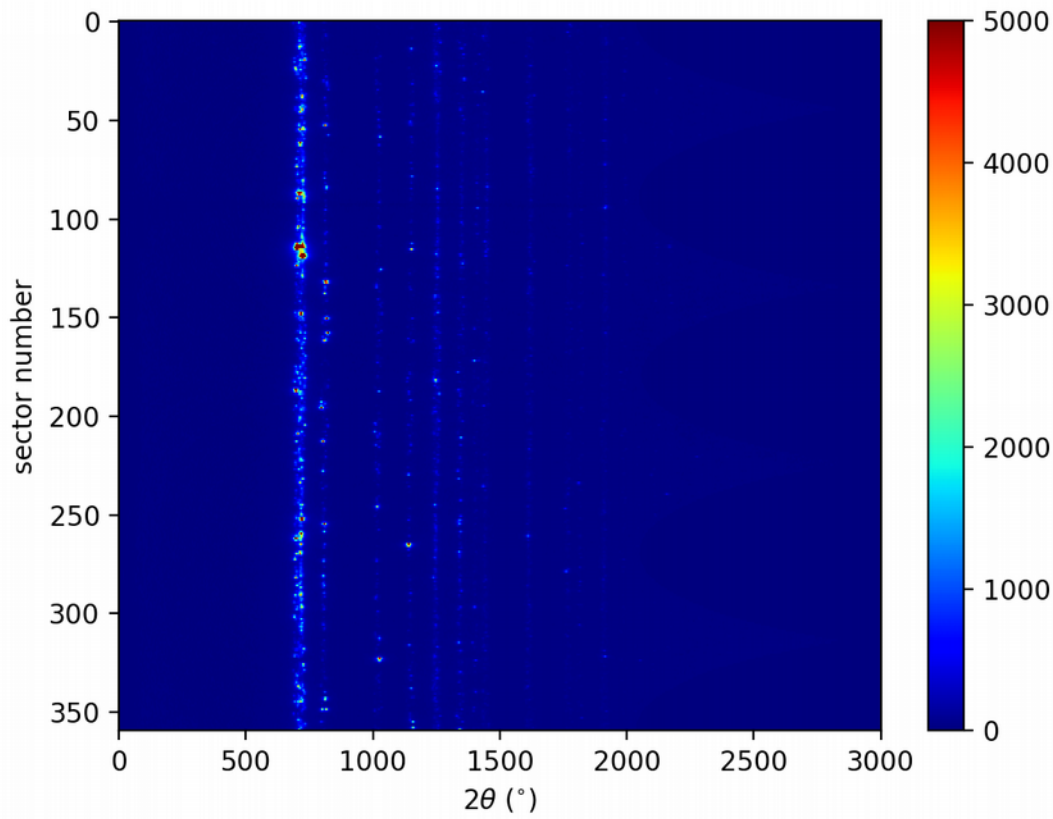
Data analysis :

Shouldered peaks - **introducing errors on calculated ferrite fraction** -



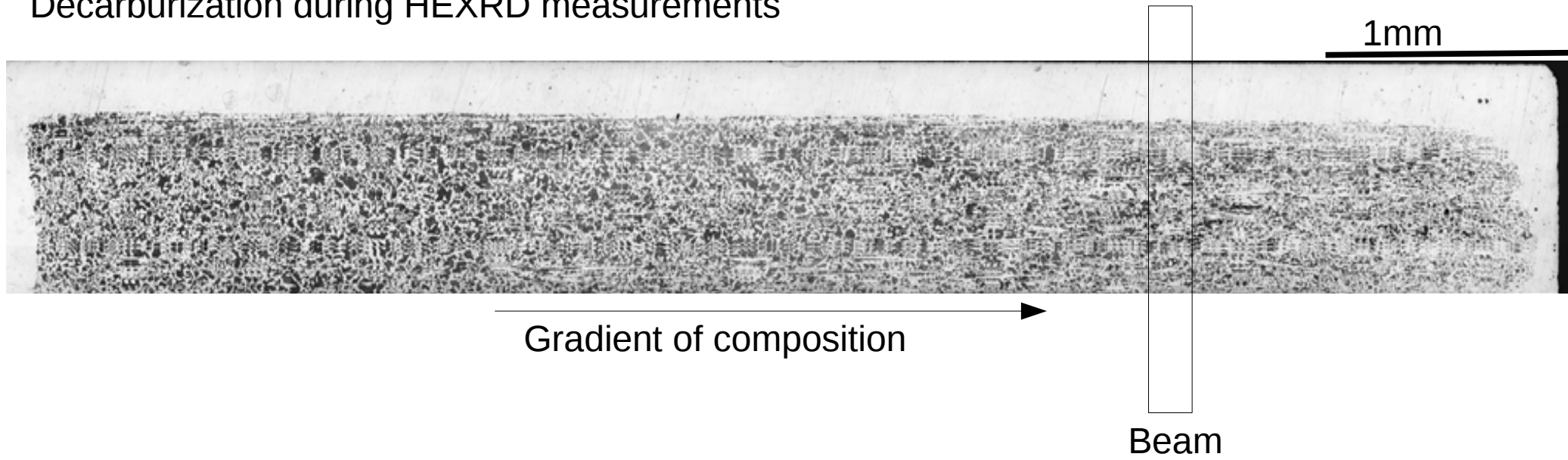
# Results

Coarse microstructure  
Saturated pixels  
No powder diffraction condition



# Results

Decarburization during HEXRD measurements





# Results

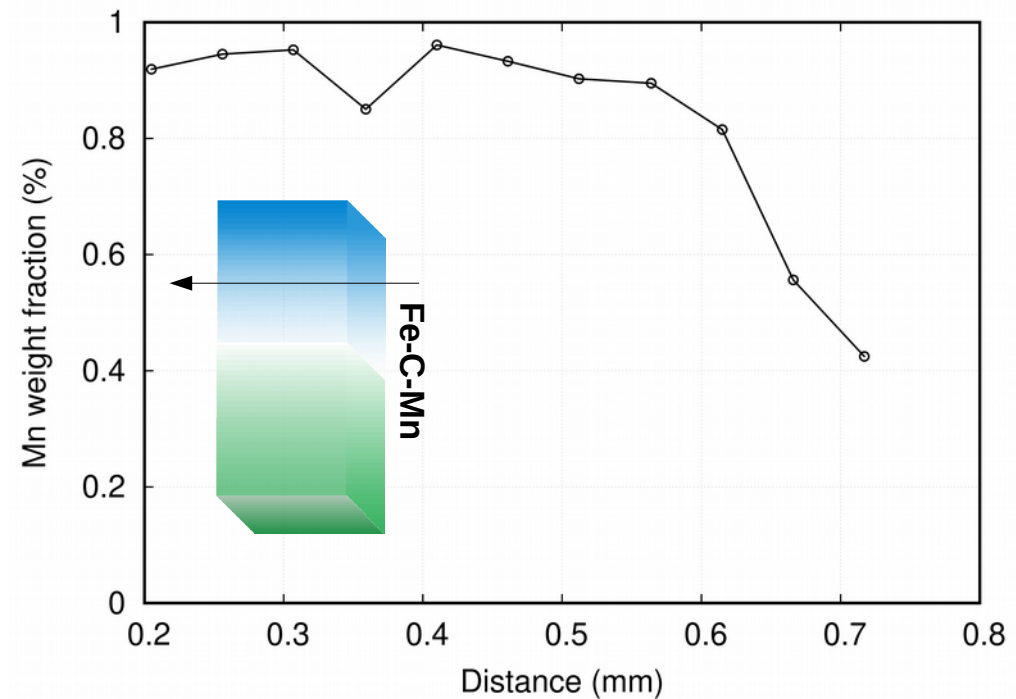
Decarburization during HEXRD measurements

1mm



Gradient of composition in the sample depth

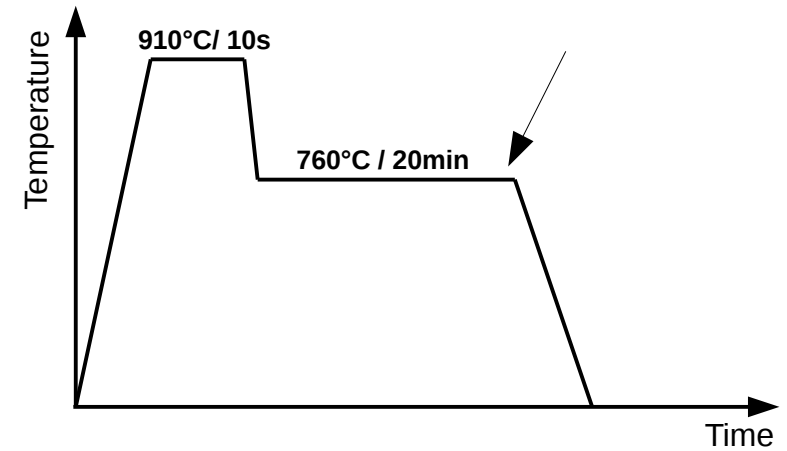
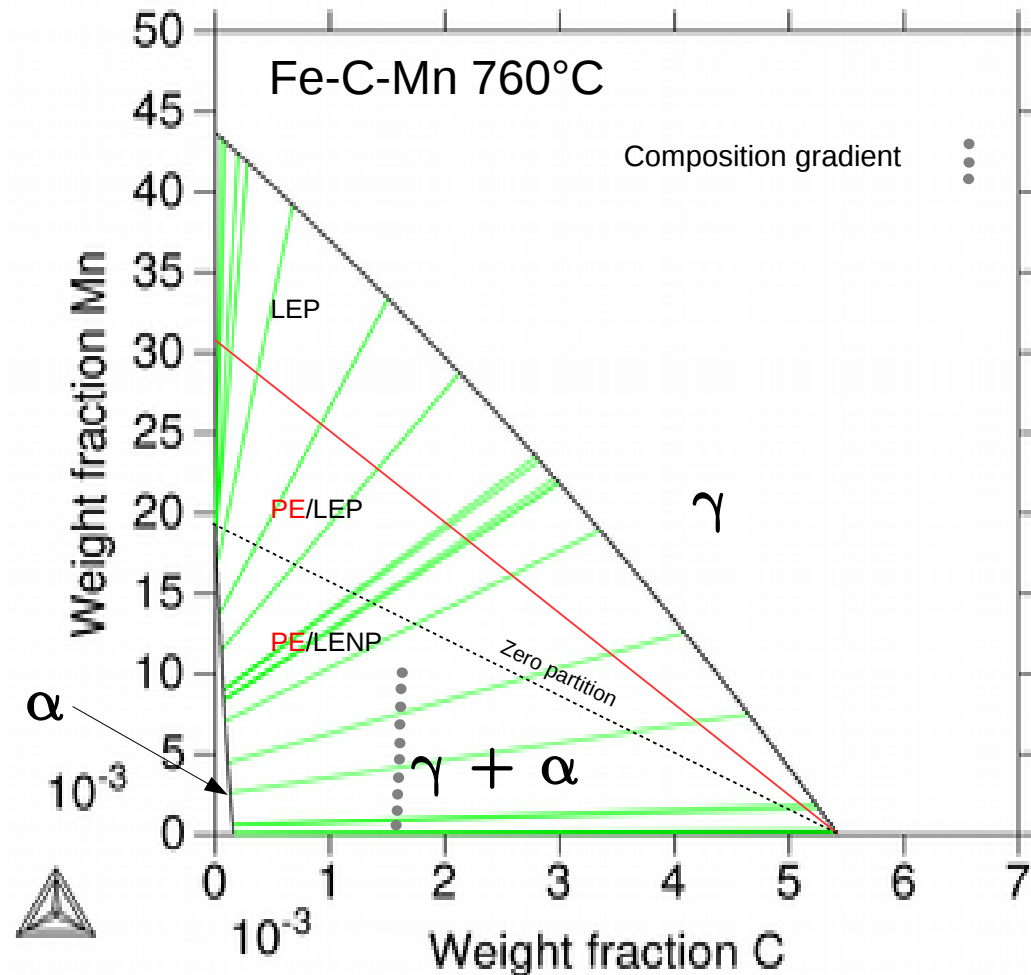
Heterogeneous deformation during cold-rolling



# Results : Effect of Mn on formed ferrite fraction

Diffusion couple : Fe-0.16C / Fe-0.16C-1Mn (%wt.)

Temperature of transformation : 760°C

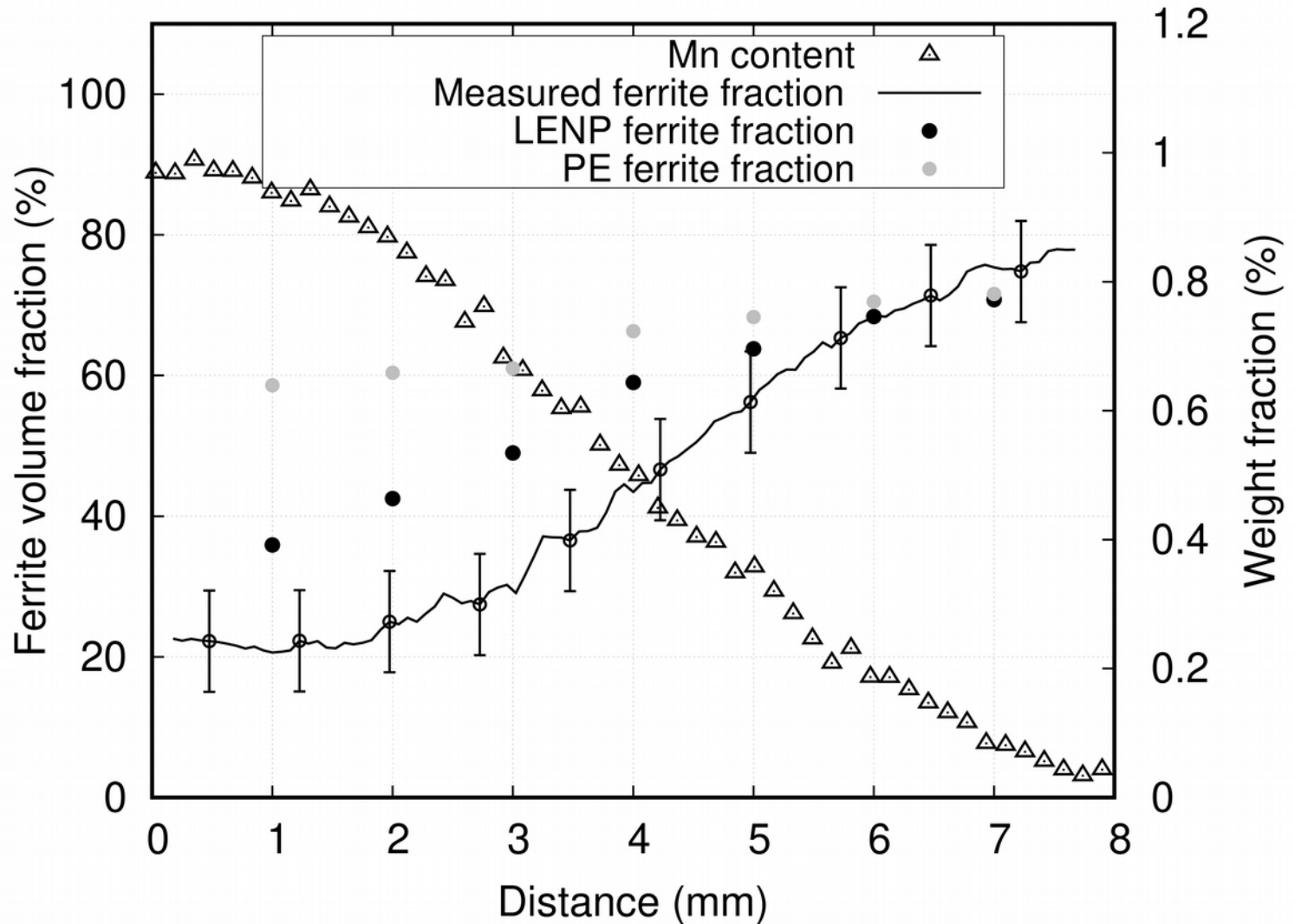
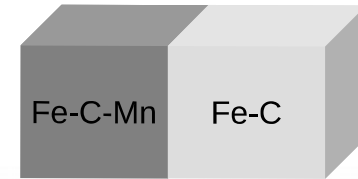


- No vertical composition gradient
- Limited decarburization 'First thermal cycle'
- Correct diffraction rings 'limited shouldered peaks effect'

# Results : Effect of Mn on formed ferrite fraction

Diffusion couple : Fe-0.16C || Fe-0.16C-1Mn (%wt.)

Temperature of transformation : 760°C after 20min



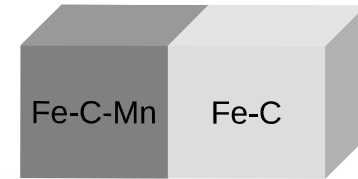
Non-complete transformation when increasing Mn content

Is it **solute drag** effect ?

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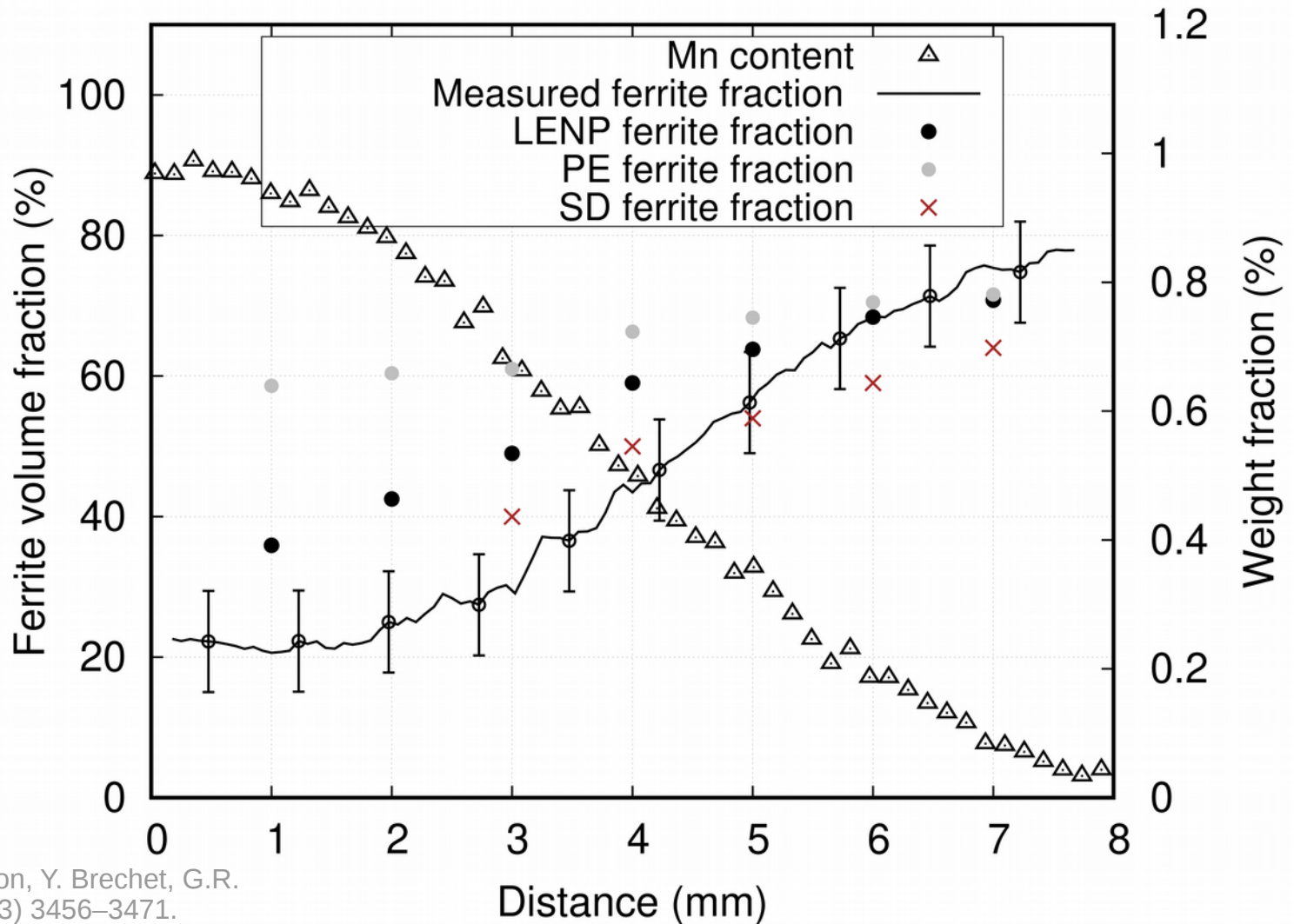
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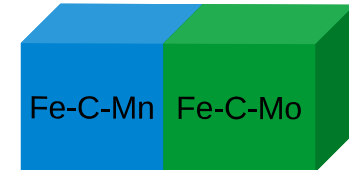


H.S. Zurob, D. Panahi, C.R. Hutchinson, Y. Brechet, G.R. Purdy, Metall. Mater. Trans. A 44 (2013) 3456–3471.

# Effect of Mn and Mo on ferrite growth kinetics

Diffusion couple

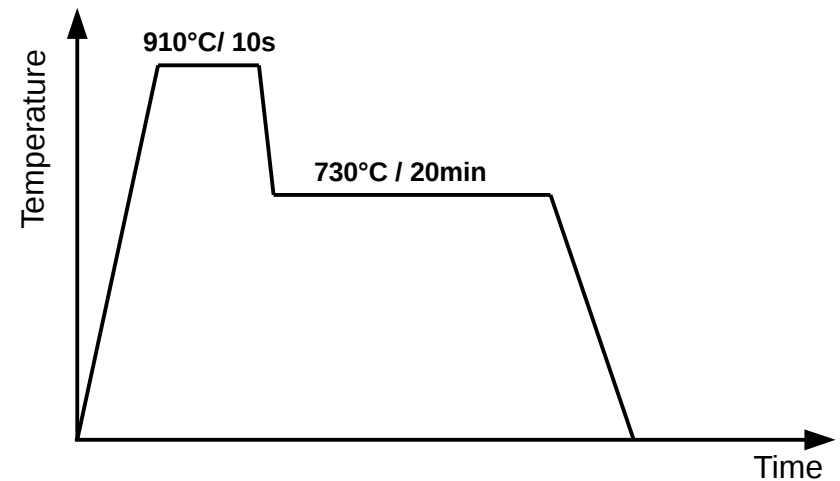
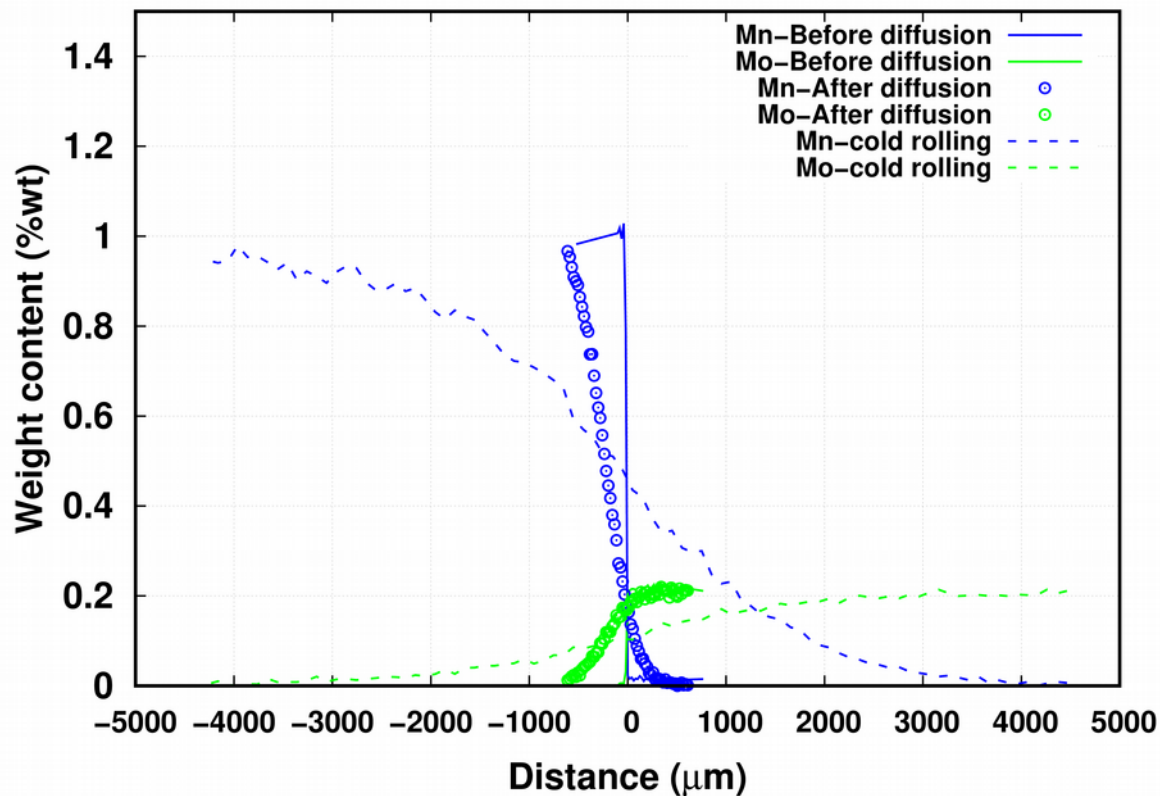
Fe-0.3%C-1%Mn || Fe-0.3%C-0.2%Mo



Profile length After cold rolling

~7 mm (Mn profile)

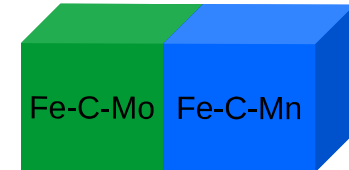
~4.5mm (Mo profile)



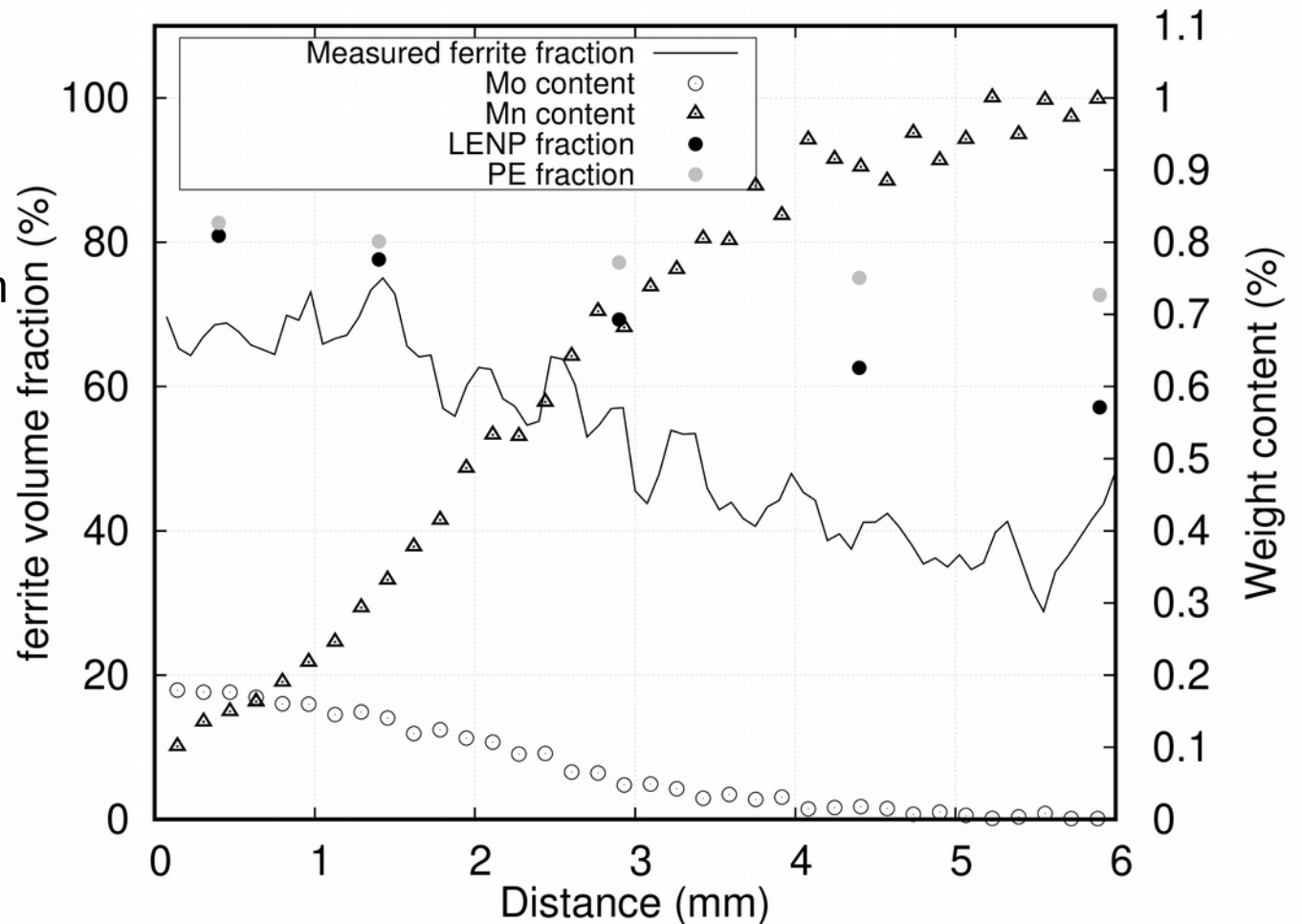
# Effect of Mn and Mo on ferrite growth kinetics

Diffusion couple Fe-0.3%C-0.2%Mo || Fe-0.3%C-1%Mn

Temperature of transformation : 730°C after 20 min

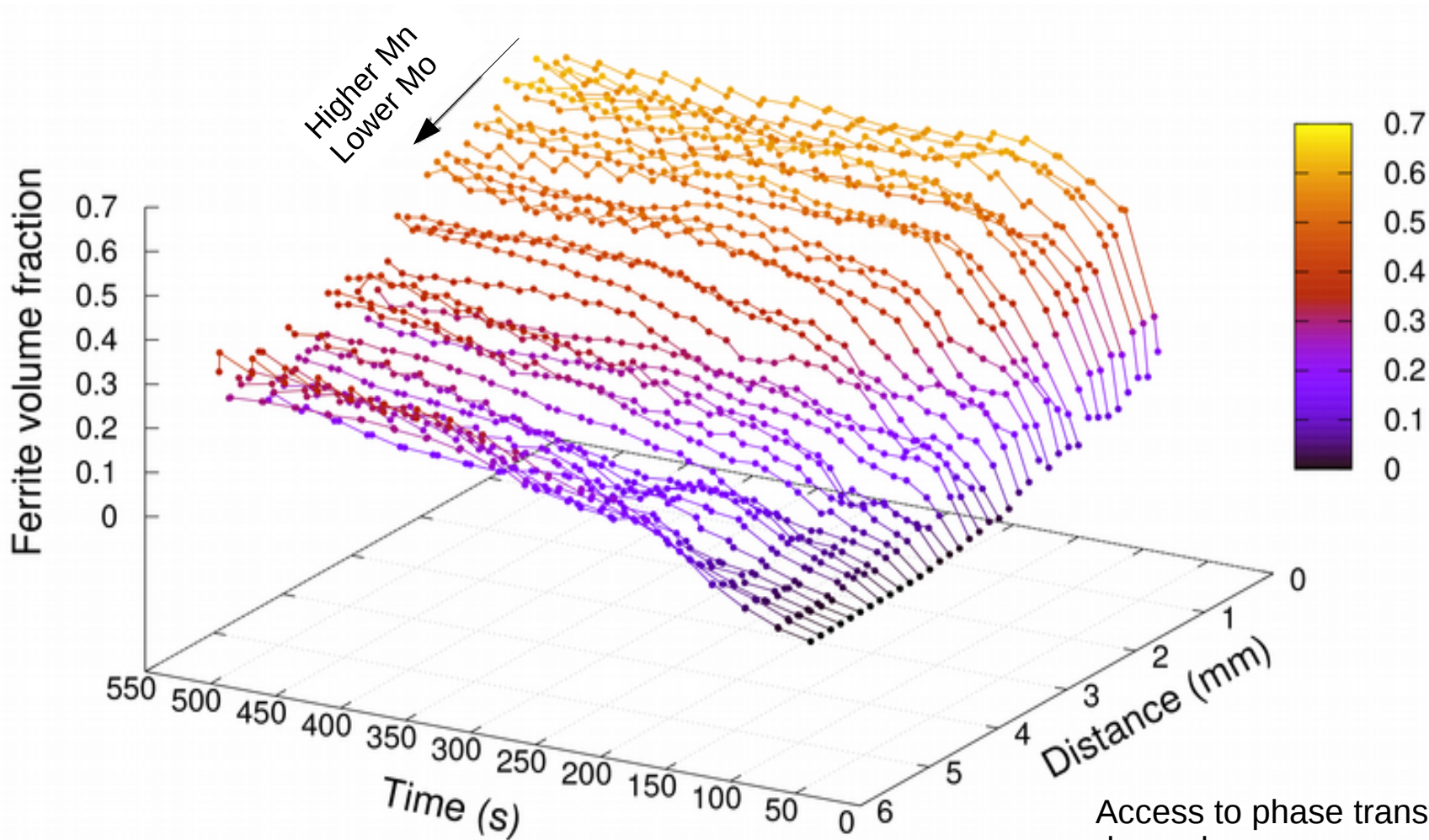


Non-complete transformation  
Increasing with Mn content



# Effect of Mn and Mo on ferrite growth kinetics

- Ferrite growth kinetics measured in a Fe-C-Mn || Fe-C-Mo diffusion couple



Access to phase transformation kinetics  
dependency on composition  
Coupling HEXRD with compositionally  
graded materials

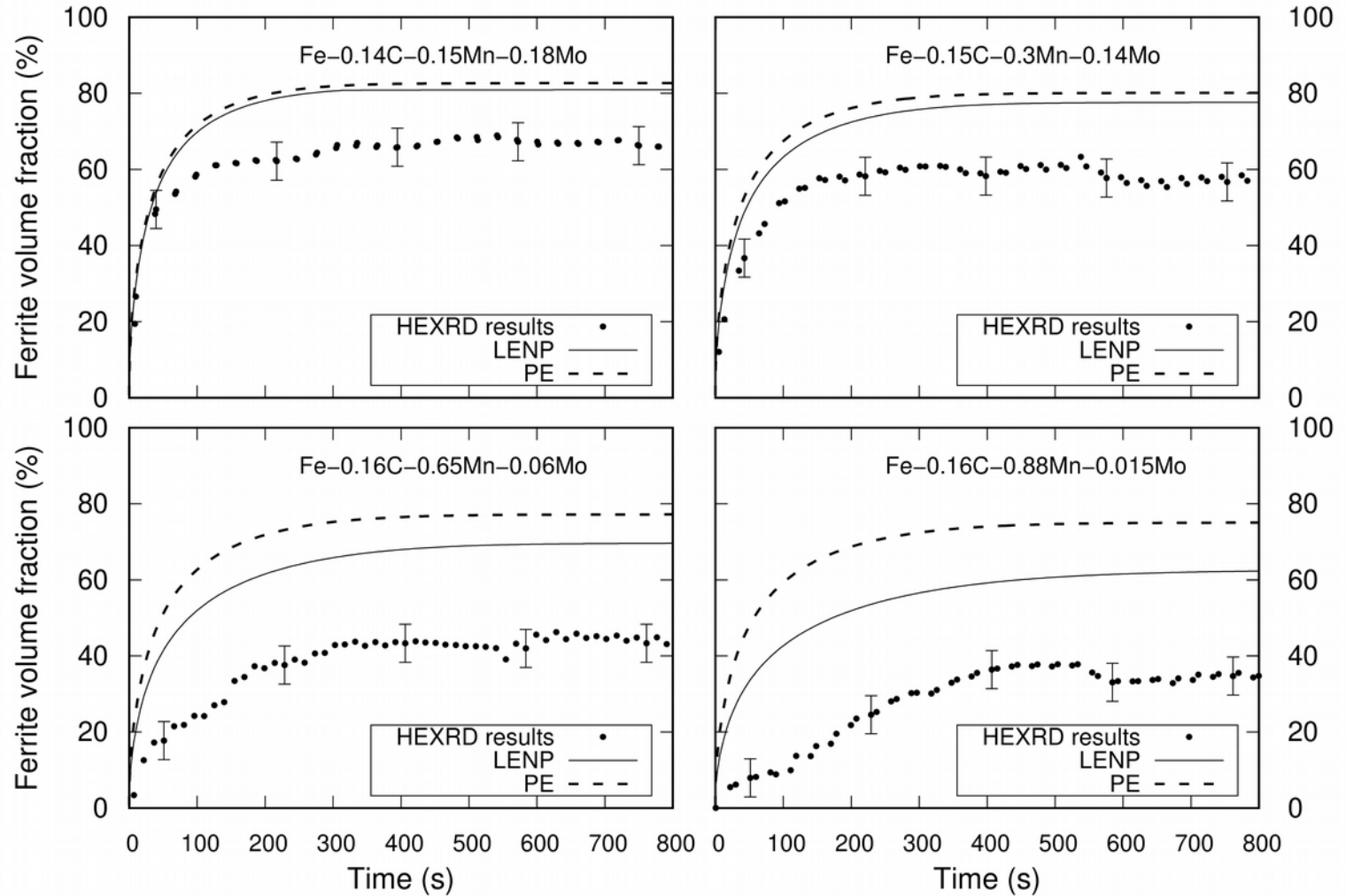
# Effect of Mn and Mo on ferrite growth kinetics

- Measured ferrite growth kinetics Vs LENP-PE predicted kinetics

Measured kinetics are slower than both PE and LENP modes

Gibbs energy dissipation by Solute-interface interaction...

Is it **Solute Drag** effect





# Effect of Mn and Mo on ferrite growth kinetics

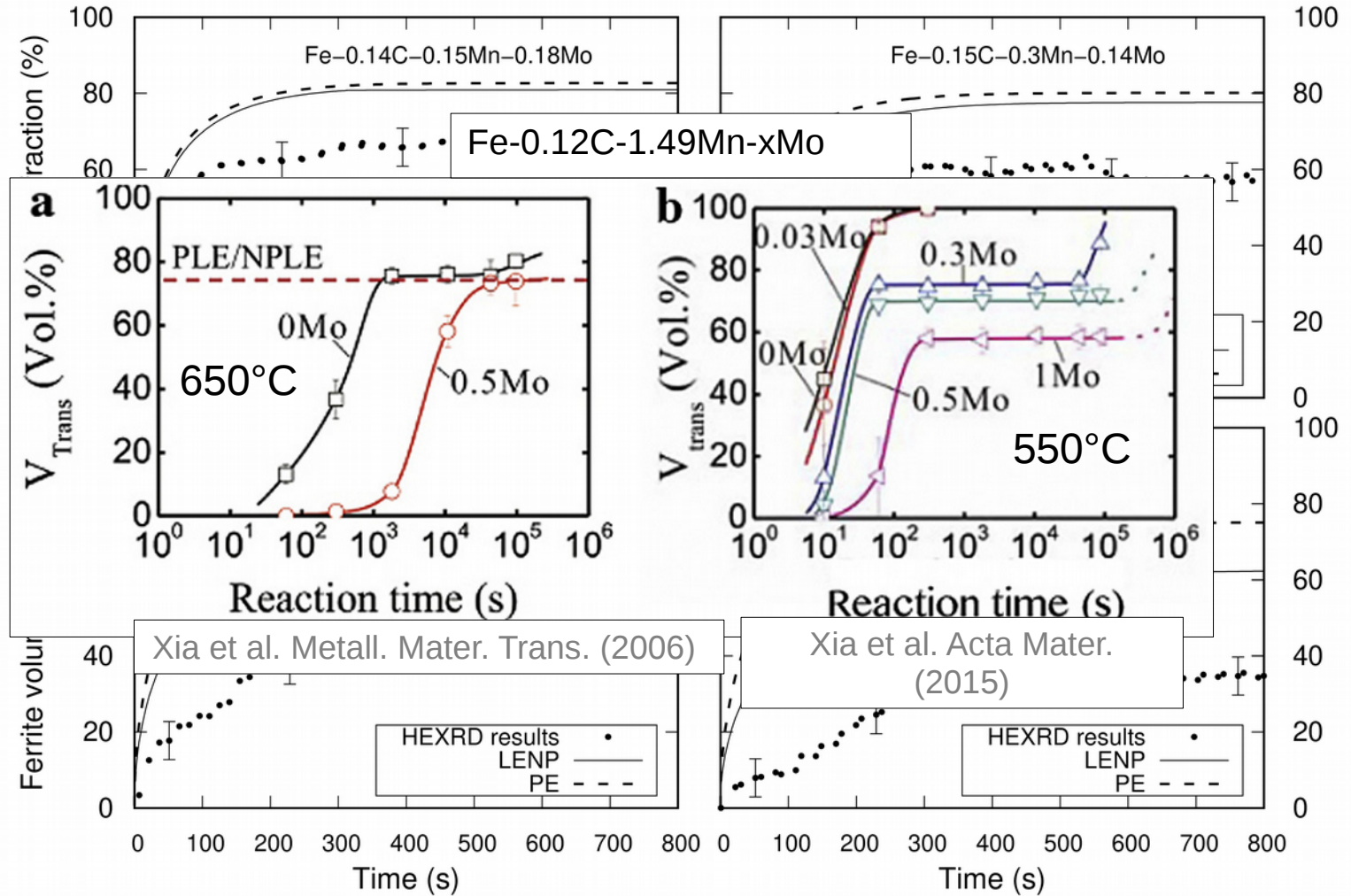
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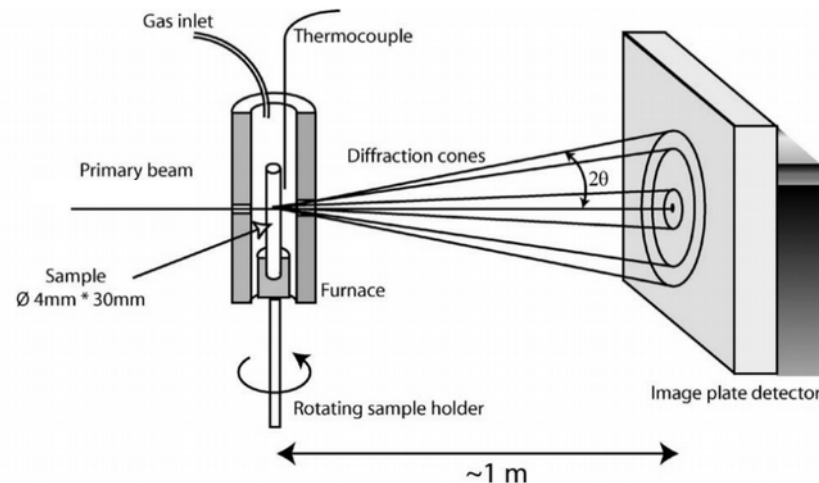
Is it **Solute Drag** effect

Retardation effect  
In Mn rich side



# What's next ?

- Implementing a new methodology for the fabrication of **compositionally-graded materials**.
- Fix the prior problems :
  - Furnace with a rotating sample holder → Grain size
  - And controlled atmosphere → Decarburization
  - Larger reduction using hot rolling followed by cold-rolling → Vertical gradient



*Guillaume Geandier  
Benoit Denand  
Intsitut Jean Lamour Nancy, France*

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- Fix the prior problems :
  - Furnace with a rotating sample holder → Grain size
  - And a better control of the atmosphere → Decarburization
  - Larger reduction using hot rolling followed by cold-rolling → Vertical gradient

Performing '**AGAIN**' time- and space-resolved high energy X-ray diffraction experiments to gather phase transformation kinetics.

Desy-Hambourg

November 2019

12 shifts

- APT (atom probe tomography) and TEM (transmission electron microscopy) techniques will be used on the treated samples to investigate the **segregation at the migrating interface**.

Thank You