# A combinatorial approach for the investigation of ferrite growth kinetics in high order systems

BENRABAH Imed-Eddine
Phd thesis : started October 2017

Guided by : Hugo Van Landeghem
Alexis Deschamps
Frederic Bonnet

Institut National Polytechnique de Grenoble
SIMAP Laboratory

ALEMI, $5^{th}$-$6^{th}$ July 2018, Metz, France.

# Introduction-motivations

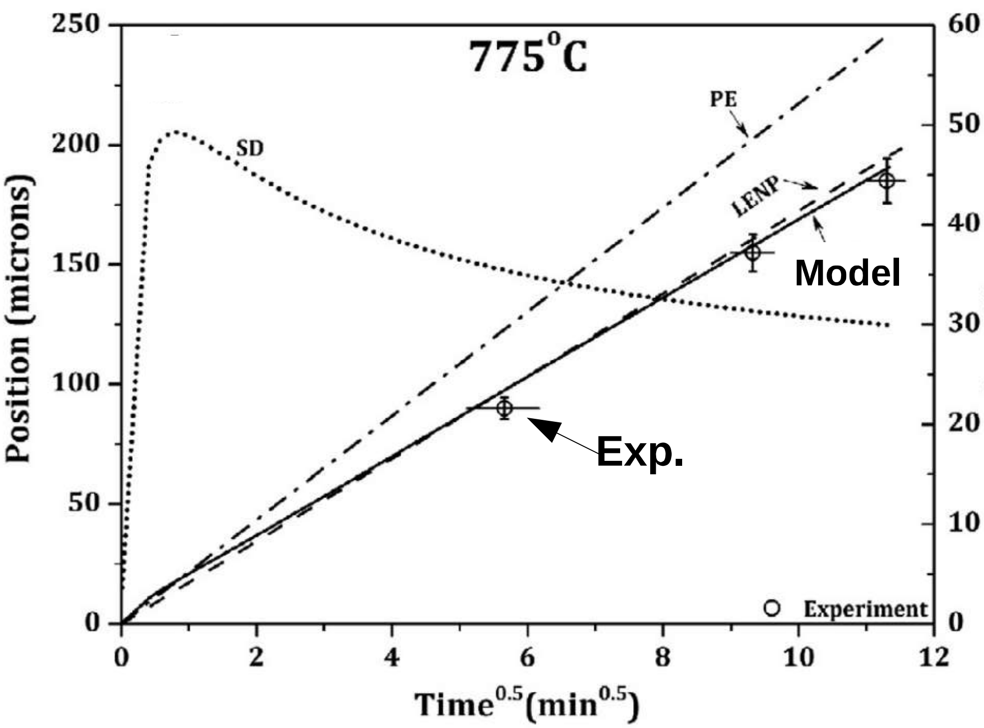
- Understanding and modeling of the kinetics of ferrite growth in multicomponent systems (Fe-C-X-Y-Z).

# Introduction-motivations

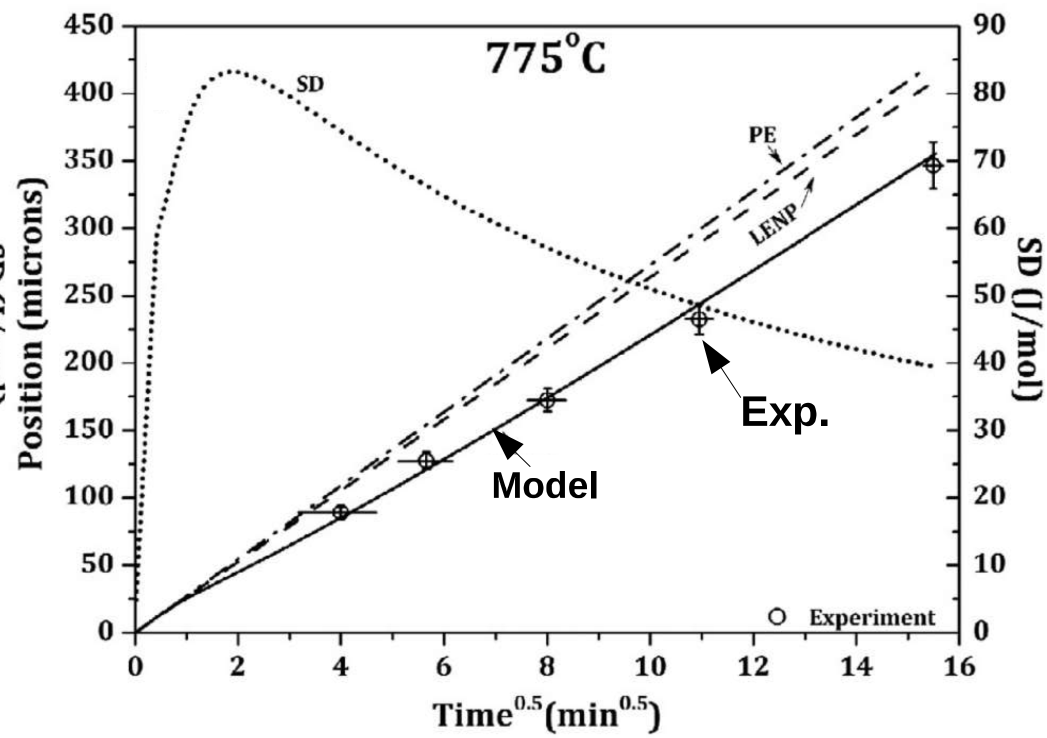
- Understanding and modeling of the kinetics of ferrite growth in multicomponent systems (Fe-C-X-Y-Z).

  - Solute Drag based models for ternary systems

Fe-C-Mn




Fe-C-Si



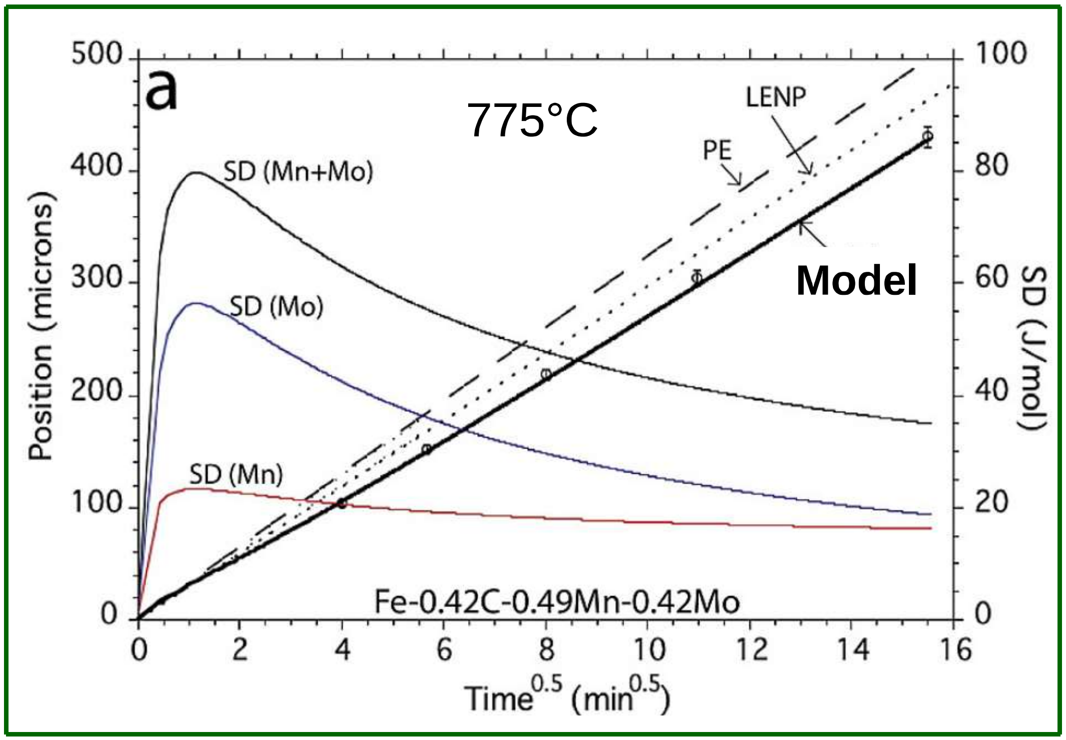

C Qiu, HS Zurob, CR Hutchinson - Acta Materialia, 2015

# Introduction-motivations

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

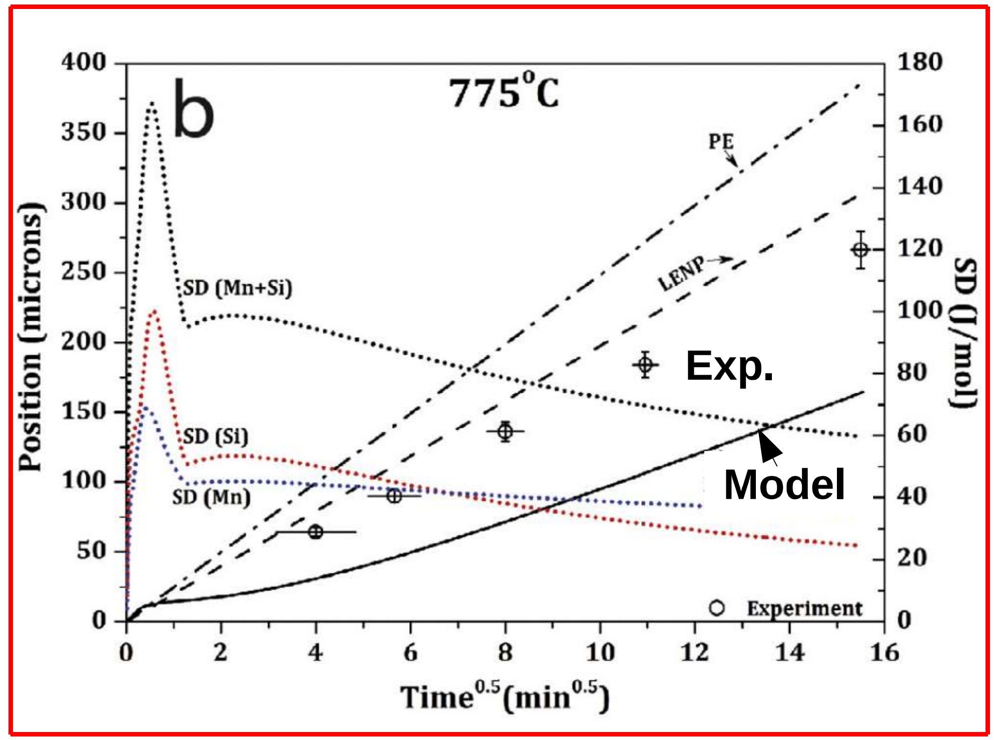
  - Solute Drag based models for Higher order systems

Fe-C-Mn-Mo




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

Fe-C-Mn-Si




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

# Introduction-motivations

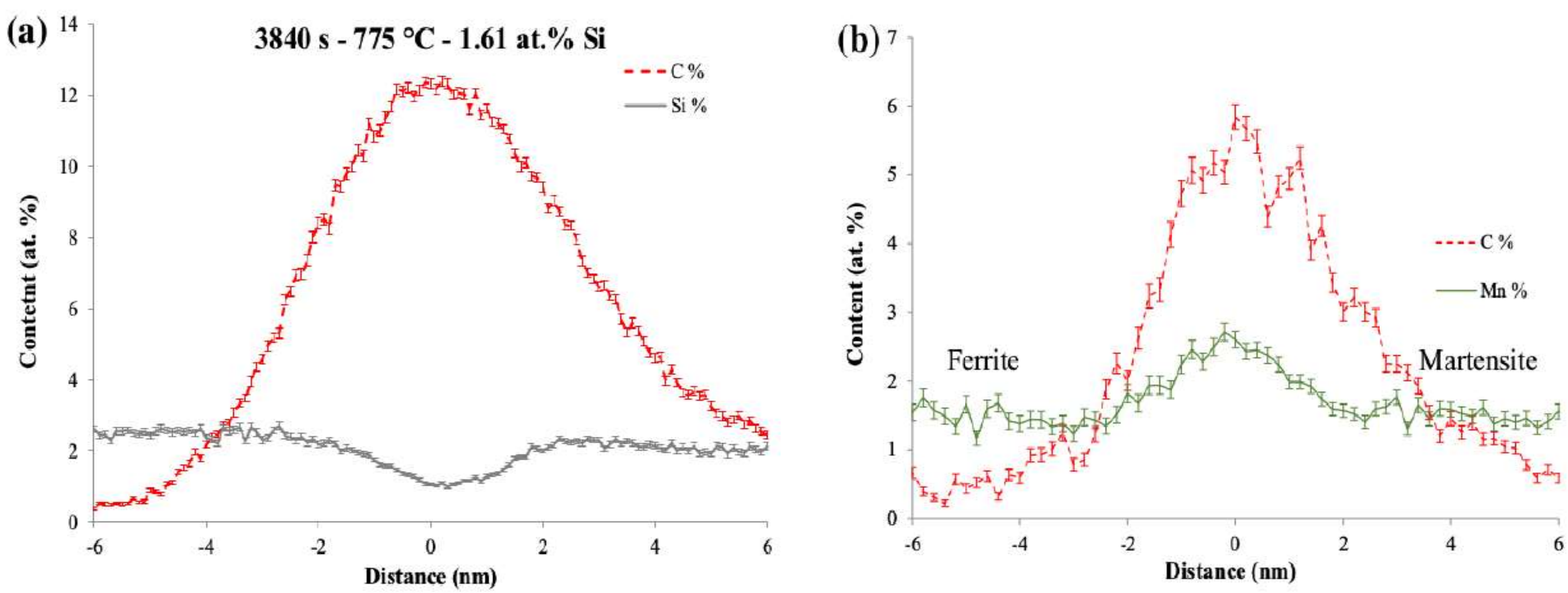
- Understanding and modeling of the kinetics of ferrite growth in multicomponent systems (Fe-C-X-Y-Z).

  - Solute segregation at the migrating interface (coupled or anti-coupled solute drag effect)

# Introduction-motivations

- 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.**

# 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.

# 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.

In one composition gradient

↓

Growth kinetics of **20** points of different compositions every **15** seconds

↓

During **300** seconds
**400** measurements per gradient

→ **Data for model calibration**

# Introduction-motivations

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

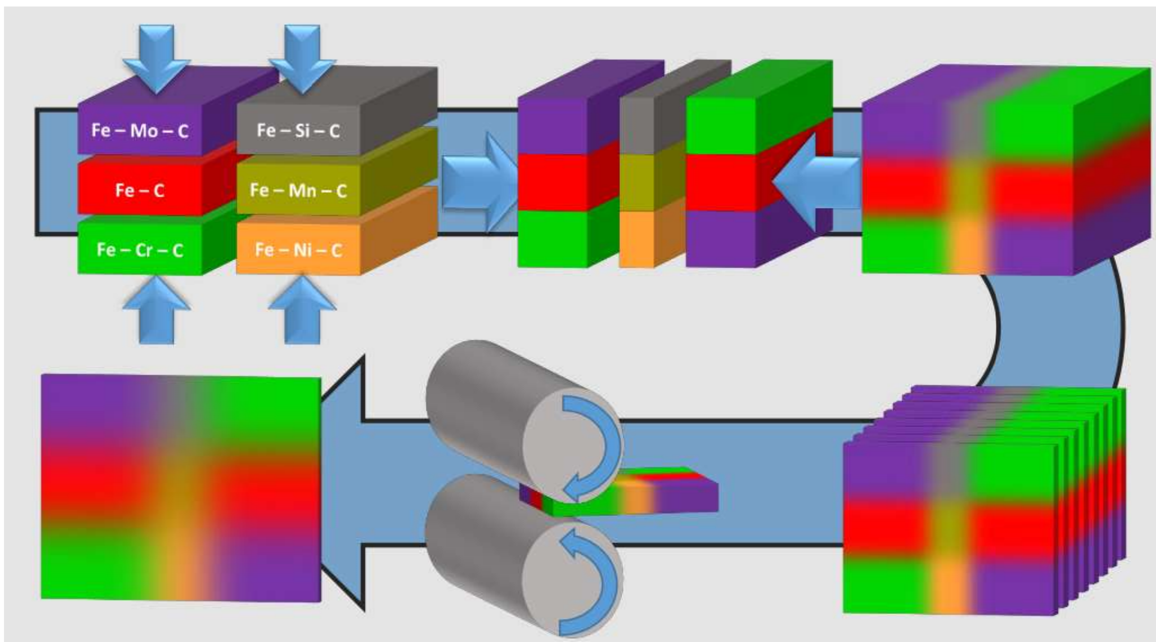
- Performing time- and space-resolved high energy X-ray diffraction experiments to gather phase transformation kinetics.

- 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



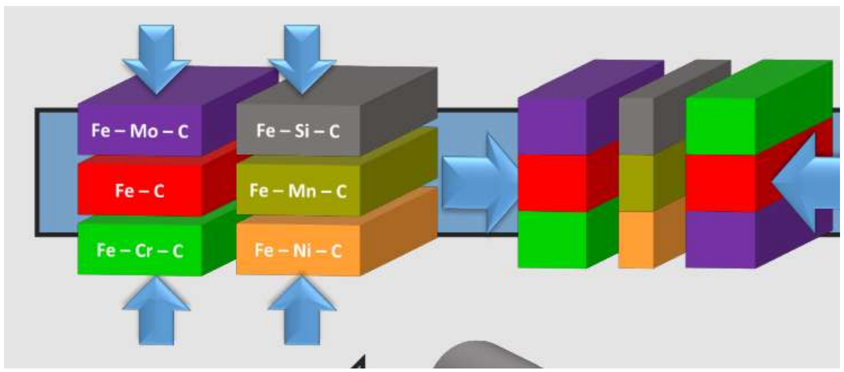

2 High temperature diffusion

3 Extending by cold/hot rolling

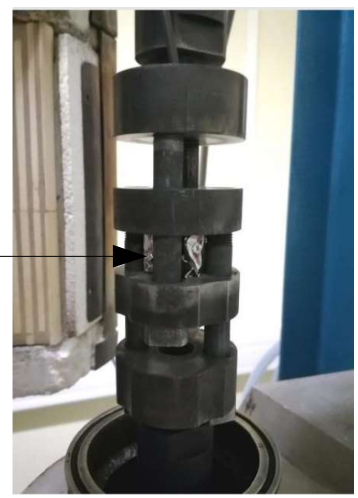
# Methodology of CGM fabrication

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

1. Solid joints
   Hot uniaxial compression



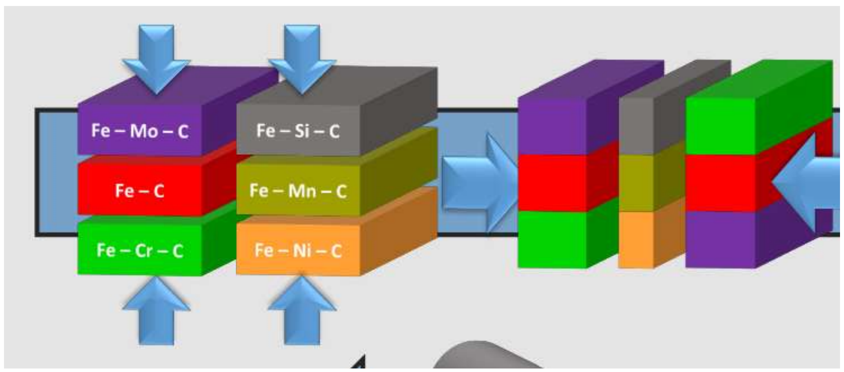

Sample



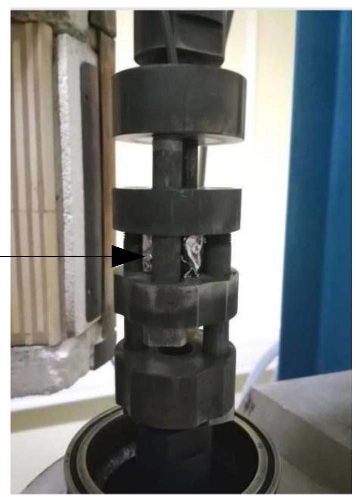
# Methodology of CGM fabrication

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

1 Solid joints
Hot uniaxial compression



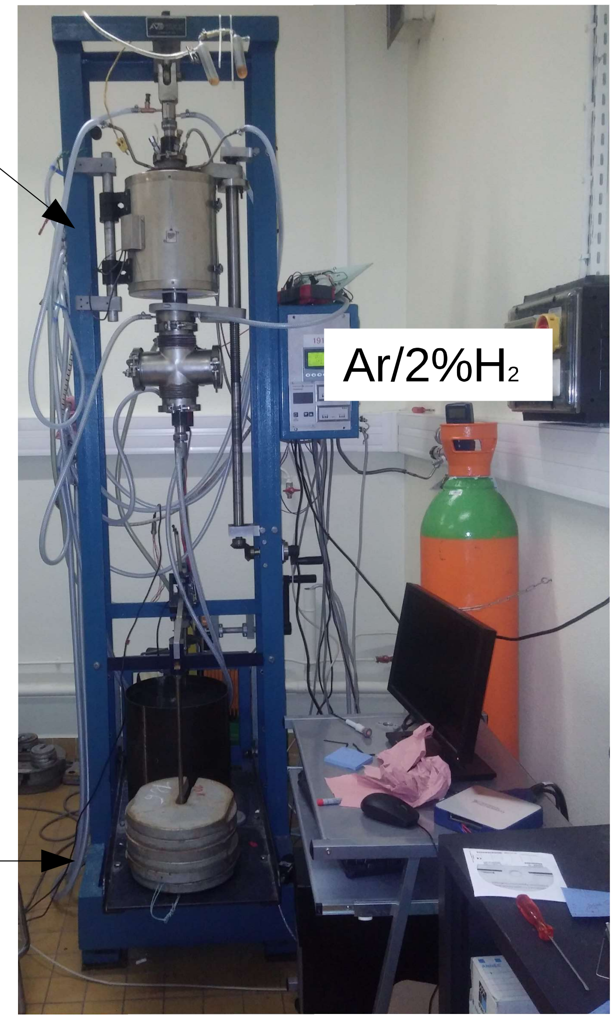

Sample



Furnace

900°C
22MPa
1hour

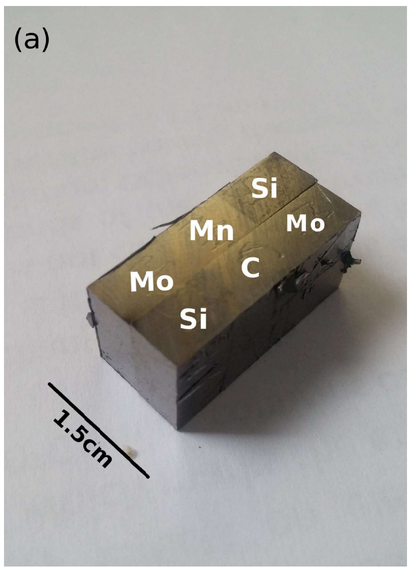
Applied weight

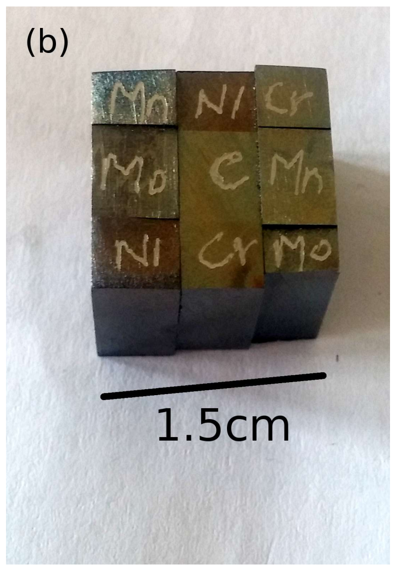


# Methodology of CGM fabrication

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

1 Solid joints
Hot uniaxial compression

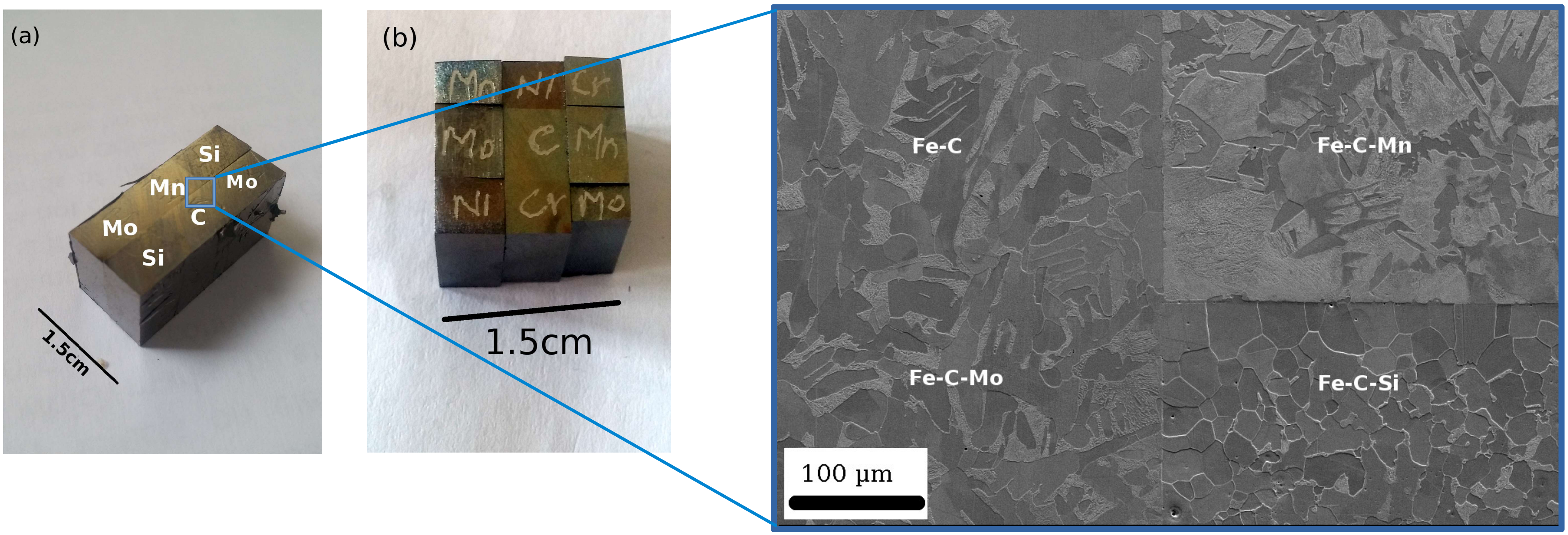


# Methodology of CGM fabrication

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

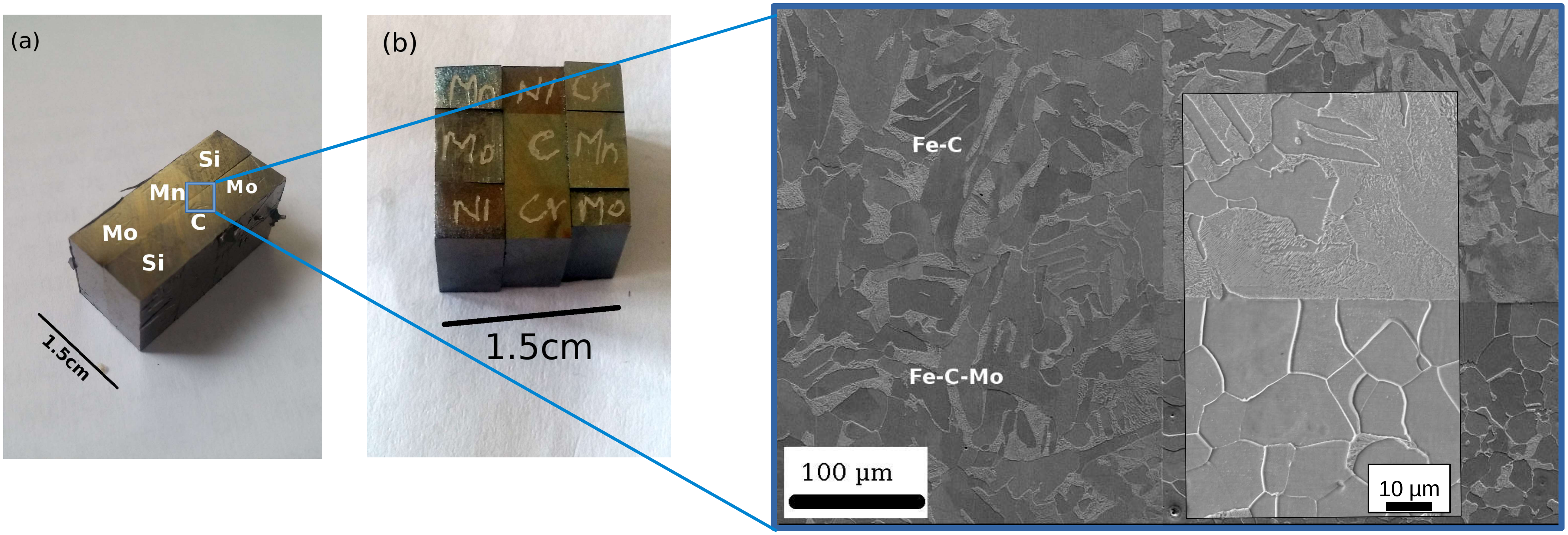
1. Solid joints
   Hot uniaxial compression



# Methodology of CGM fabrication

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

1 Solid joints
Hot uniaxial compression

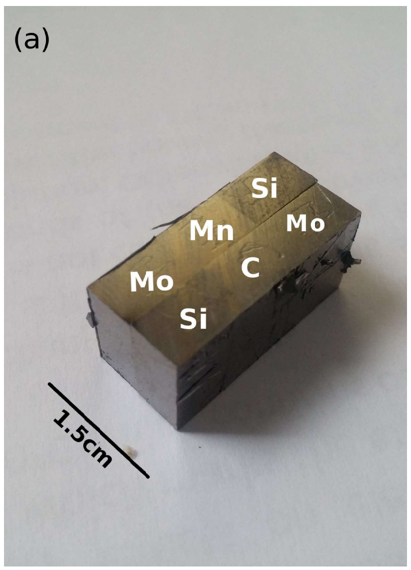


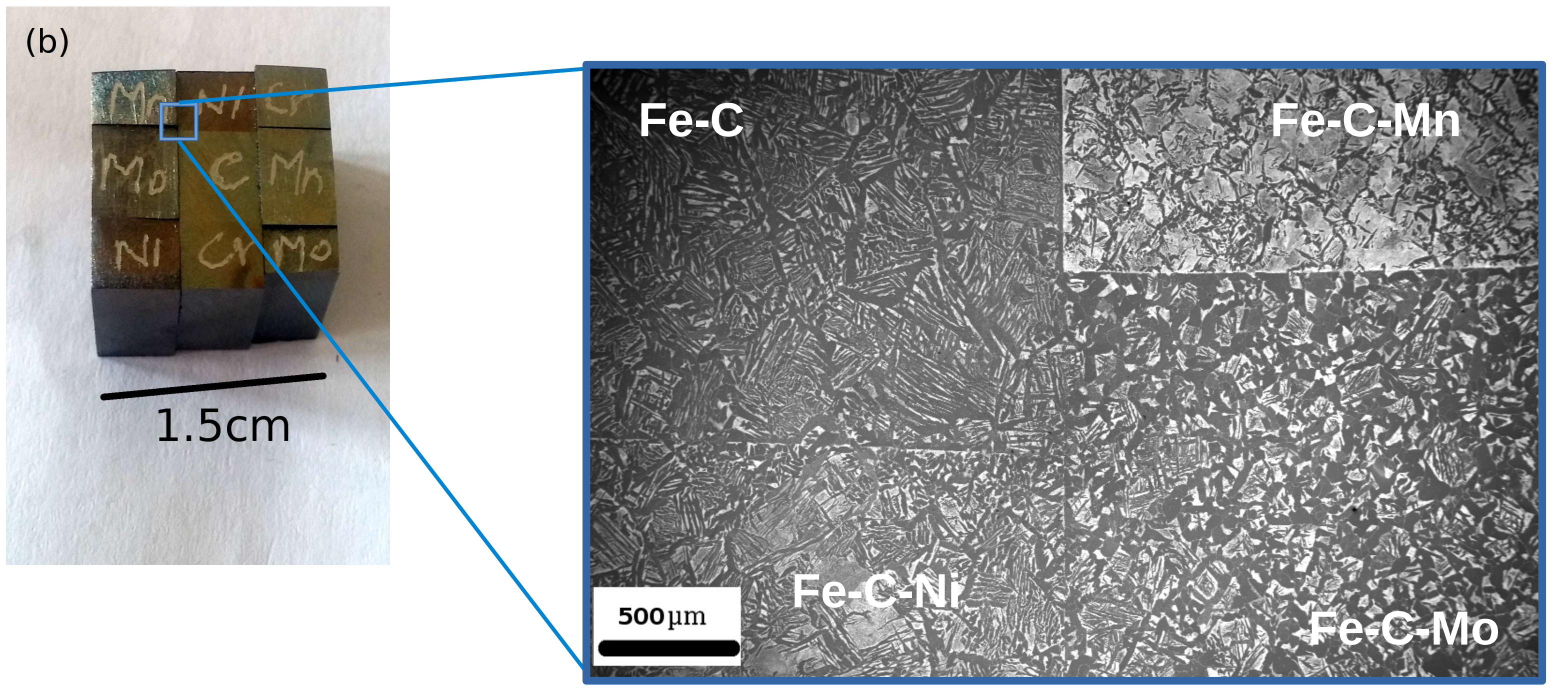

Oxide and pore-free interfaces

# Methodology of CGM fabrication

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

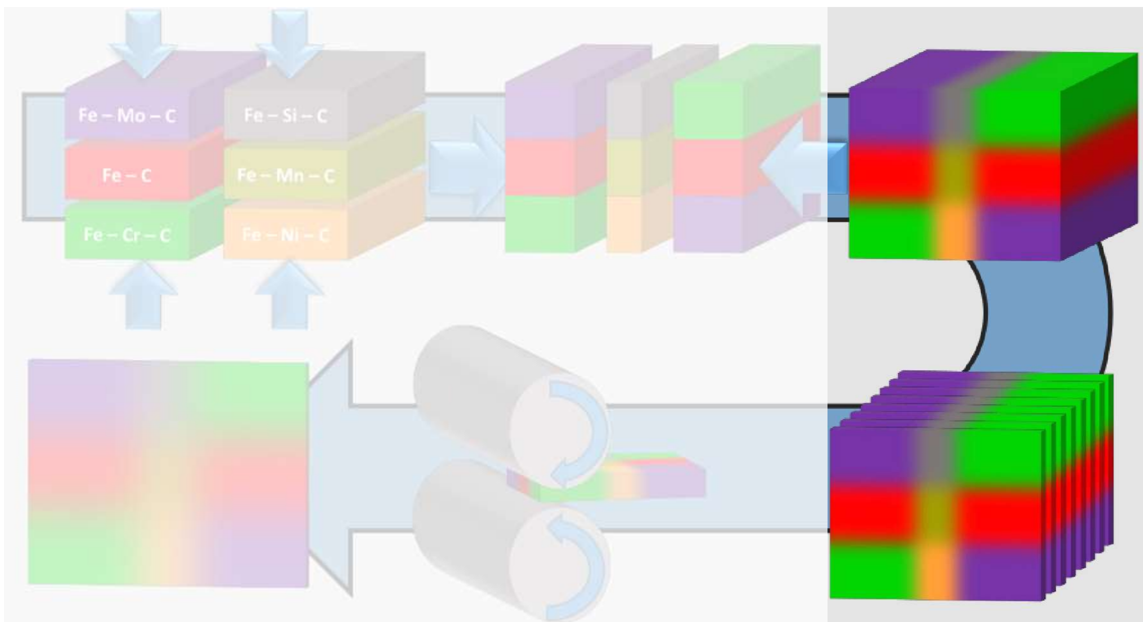
1. Solid joints
   Hot uniaxial compression



# Methodology of CGM fabrication

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




2

High temperature diffusion

# Methodology of CGM fabrication

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

2

High temperature diffusion

Generate gradients of composition

Perform diffusion in the single phase domain

1380°C for 72 hours → ~ 600-900 µm

Good control of diffusion atmosphere

Avoid decarburization and oxidation

# Methodology of CGM fabrication

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

2

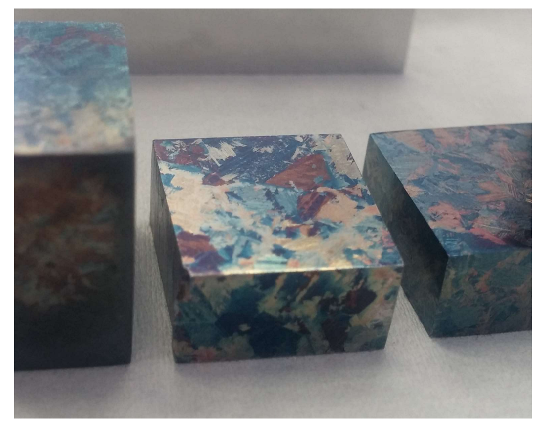
High temperature diffusion

Oxidation :

Furnace cleaning :

- Multiple Ar/$H_2$ gas purges
- Several days at high temperature (1400°C) under vacuum ($10^{-2}$ mbar)
- Keeping the furnace under vacuum and at 300°C

Total oxidation



Extreme surface oxidation

# Methodology of CGM fabrication

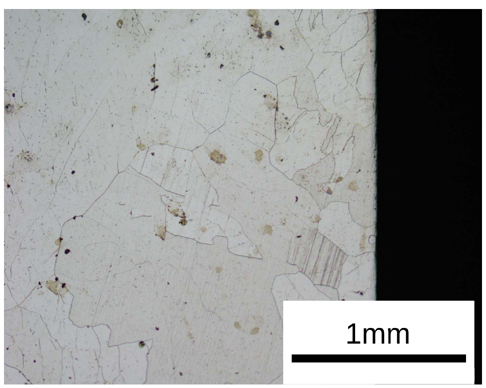
- Implementing a new methodology for the fabrication of **compositionally-graded materials.**

2

High temperature diffusion

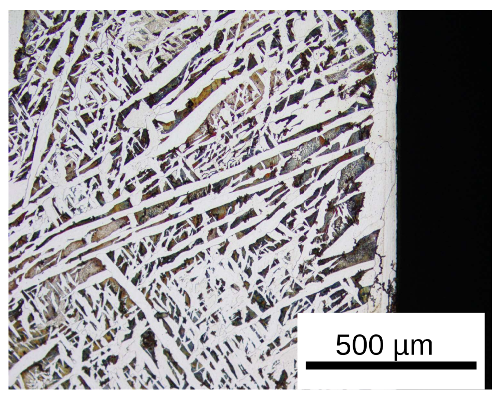
**Decarburization :** 10 hours at 1380°C

Argon (3% Hydrogen)
~2.7 mbar



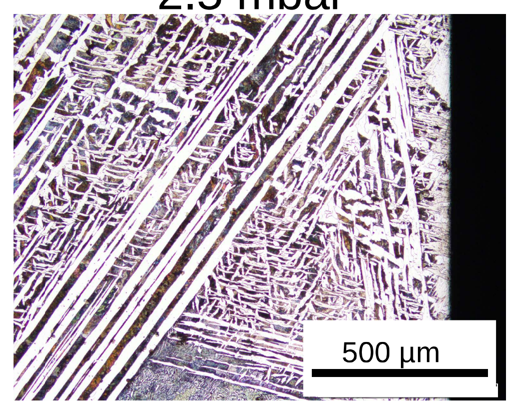

Total decarburization

Argon (0.3% Hydrogen )
~2.5 mbar




90 µm of decarburization

Argon (0% Hydrogen )
~2.5 mbar




NO decarburization

# Methodology of CGM fabrication

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

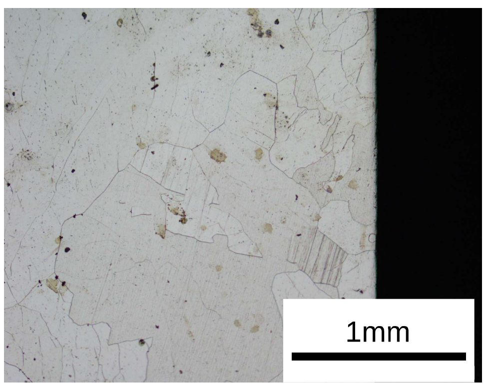
2

High temperature diffusion

Decarburization : 10 hours at 1380°C

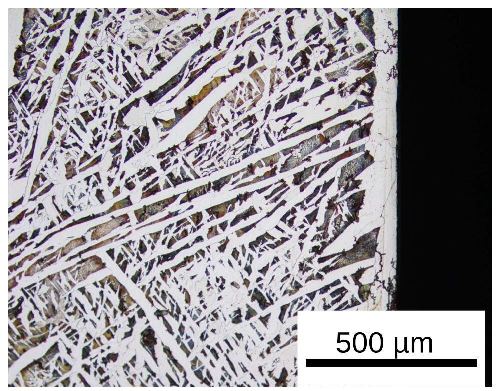
Reproducibility !!

Argon (3% Hydrogen)
2.5 mbar



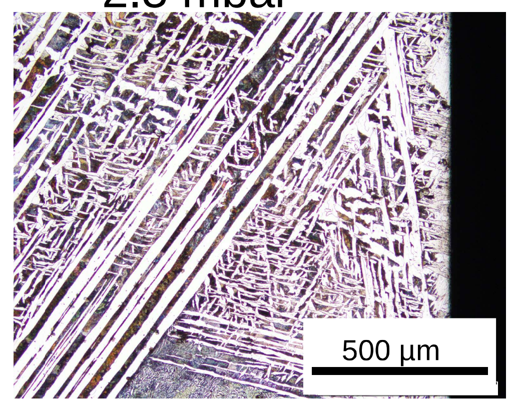

Total decarburization

Argon (0.3% Hydrogen )
2.5 mbar




90 µm of decarburization

Argon (0% Hydrogen )
2.5 mbar




NO decarburization

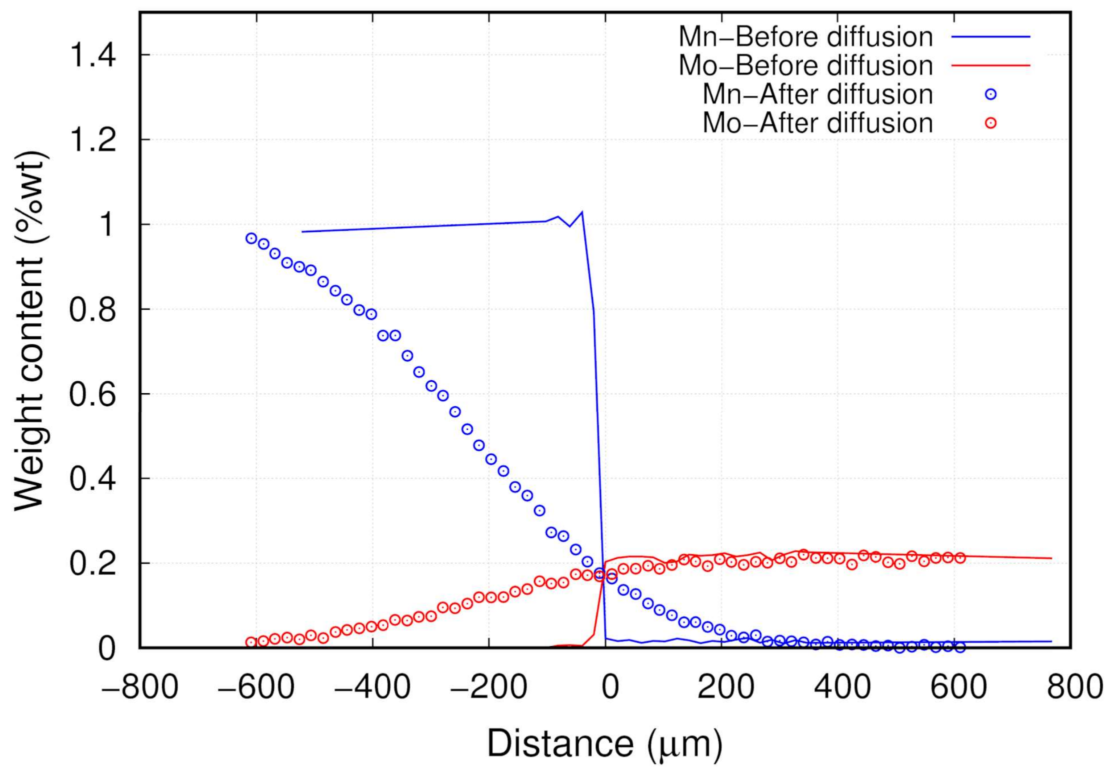
# Methodology of CGM fabrication

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

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 (0% Hydrogen)

Profile length after diffusion

950µm (Mn profile)
700µm (Mo profile)

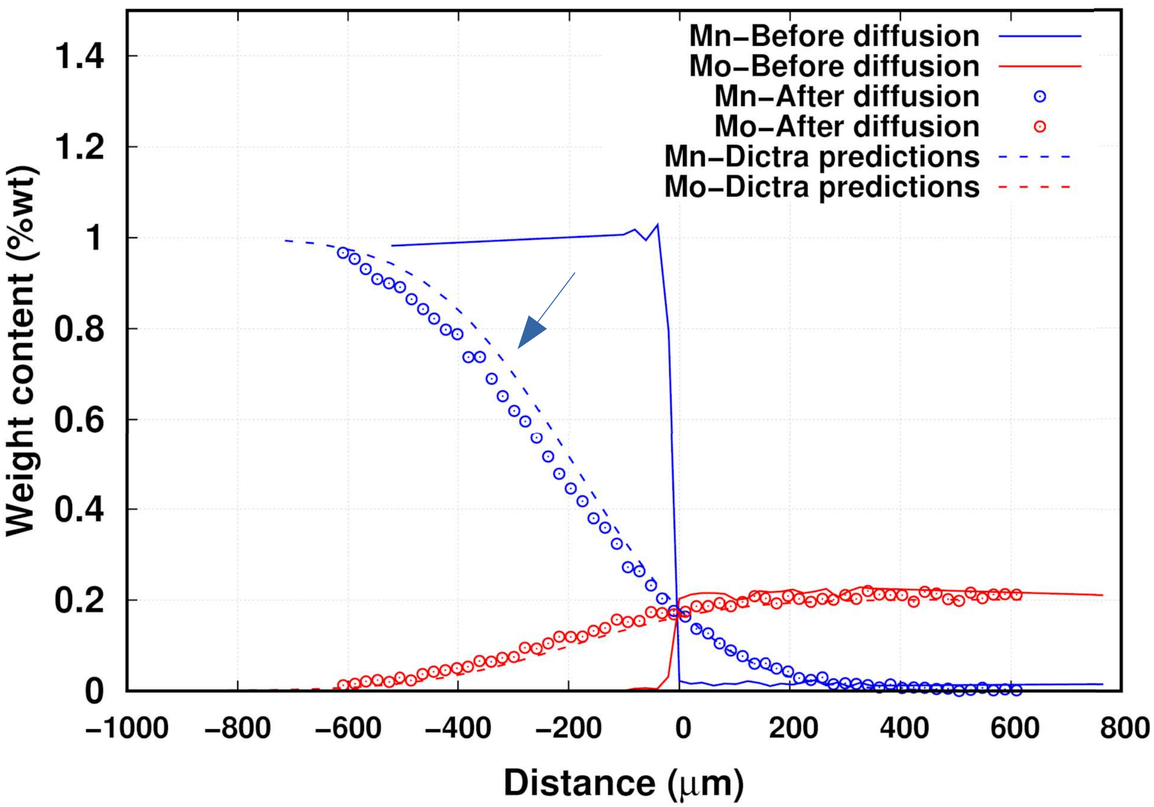
# Methodology of CGM fabrication

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

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 (0% Hydrogen)

Profile length after diffusion

950µm (Mn profile)
700µm (Mo profile)

In good agreement with DICTRA simulations

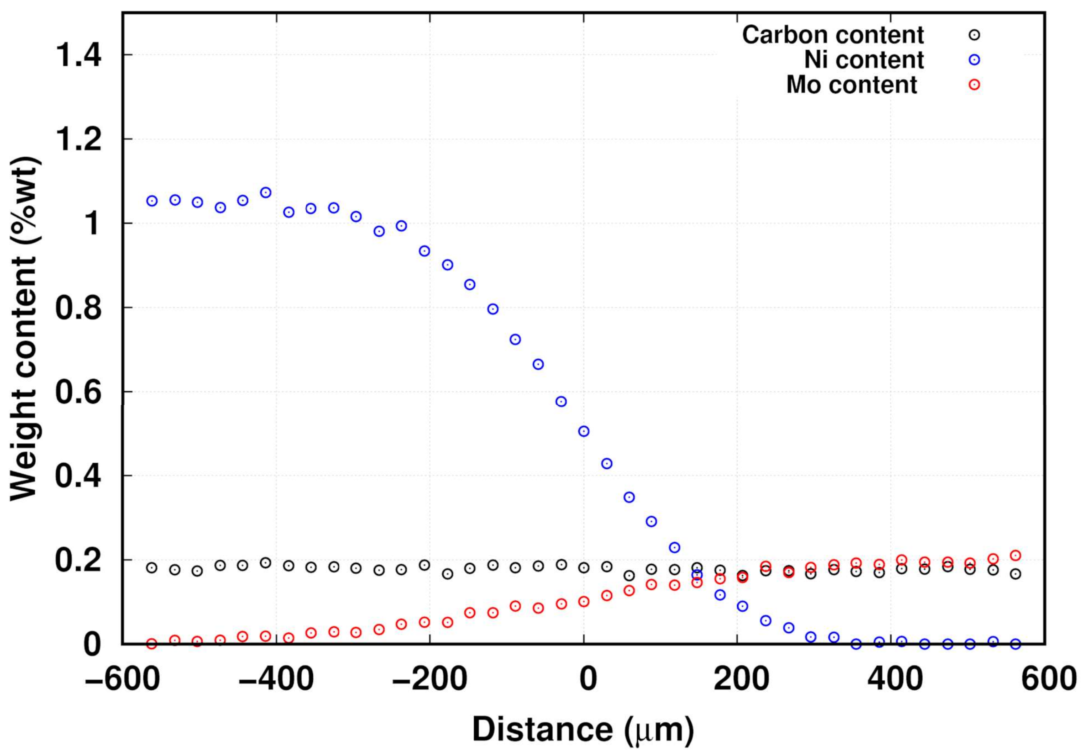
# Methodology of CGM fabrication

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

2

High temperature diffusion

EPMA measurements before and after diffusion treatment




Diffusion couple

Fe-0.3%C-1%Ni Fe-0.3%C-0.2%Mo

72 hours at 1380°C

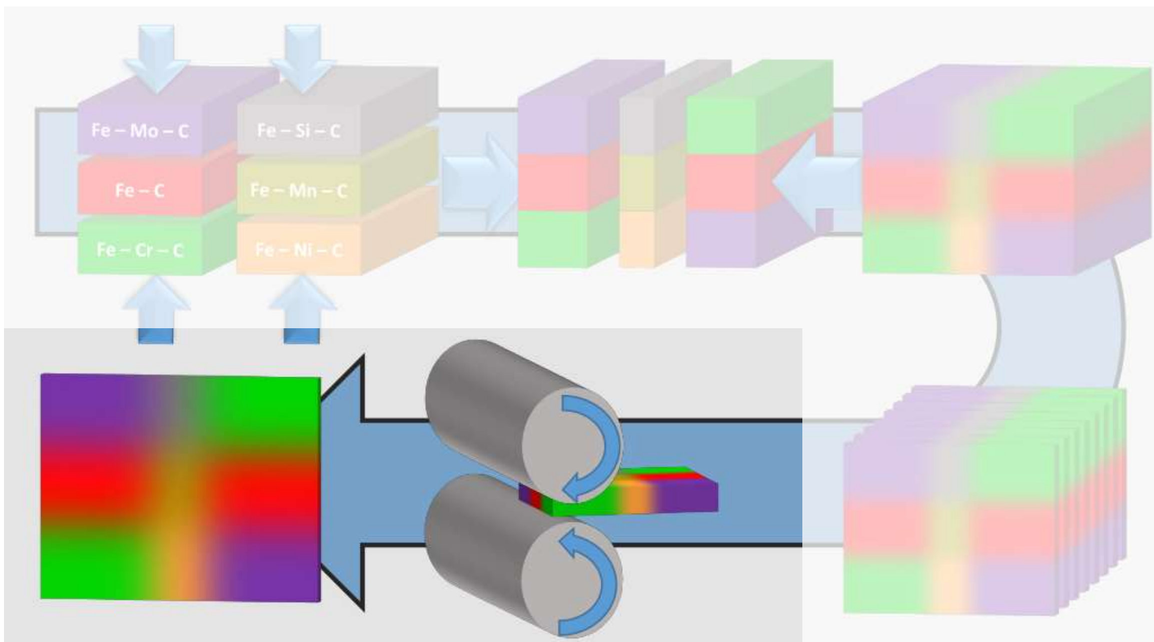
2.5mbar – Argon (0% Hydrogen)

Carbon concentration : 0.3% to 0.2%

Decarburization !!

# Methodology of CGM fabrication

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



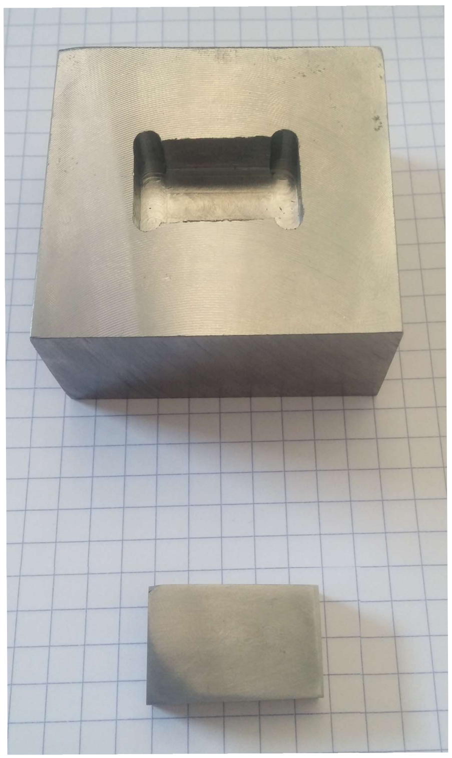

3 Extending by cold/hot rolling

# Methodology of CGM fabrication

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

3 Extending by cold/hot rolling

Low carbon steel



Sample

Reduction ~85%

Thickness : 7mm – to – 1mm

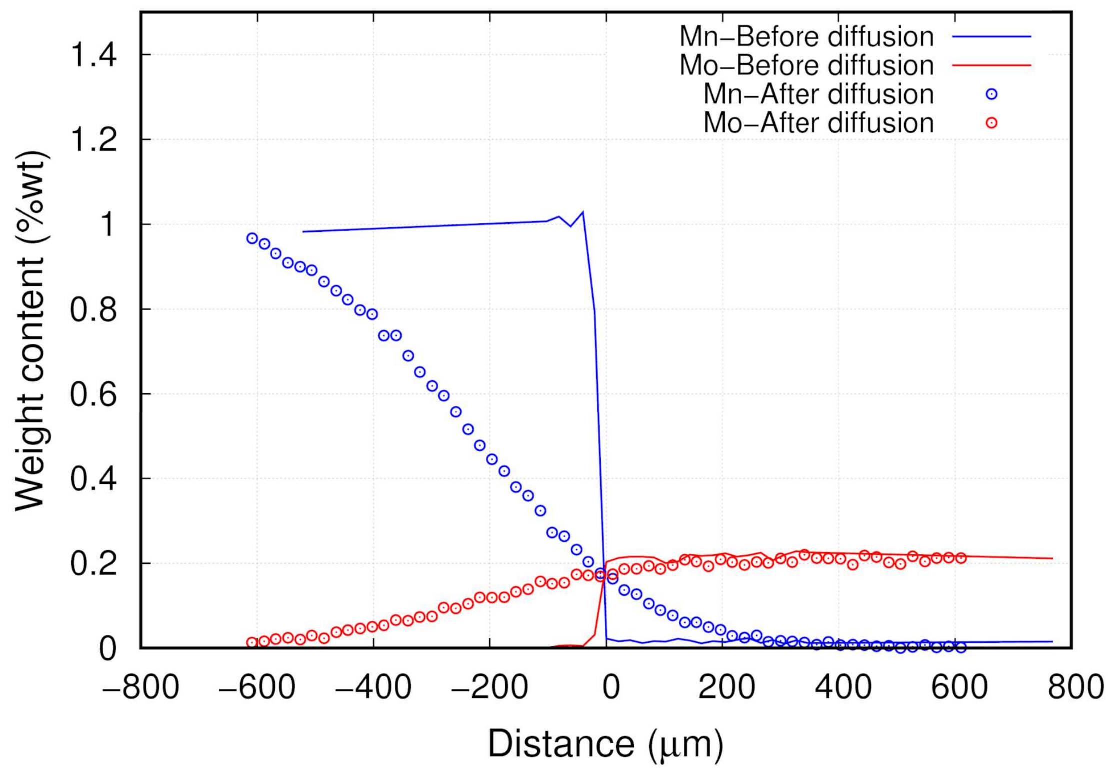
Small multiple cold rolling passes

# Methodology of CGM fabrication

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

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

72 hours at 1380°C

2.5mbar – Argon (0% Hydrogen)

Profile length after diffusion

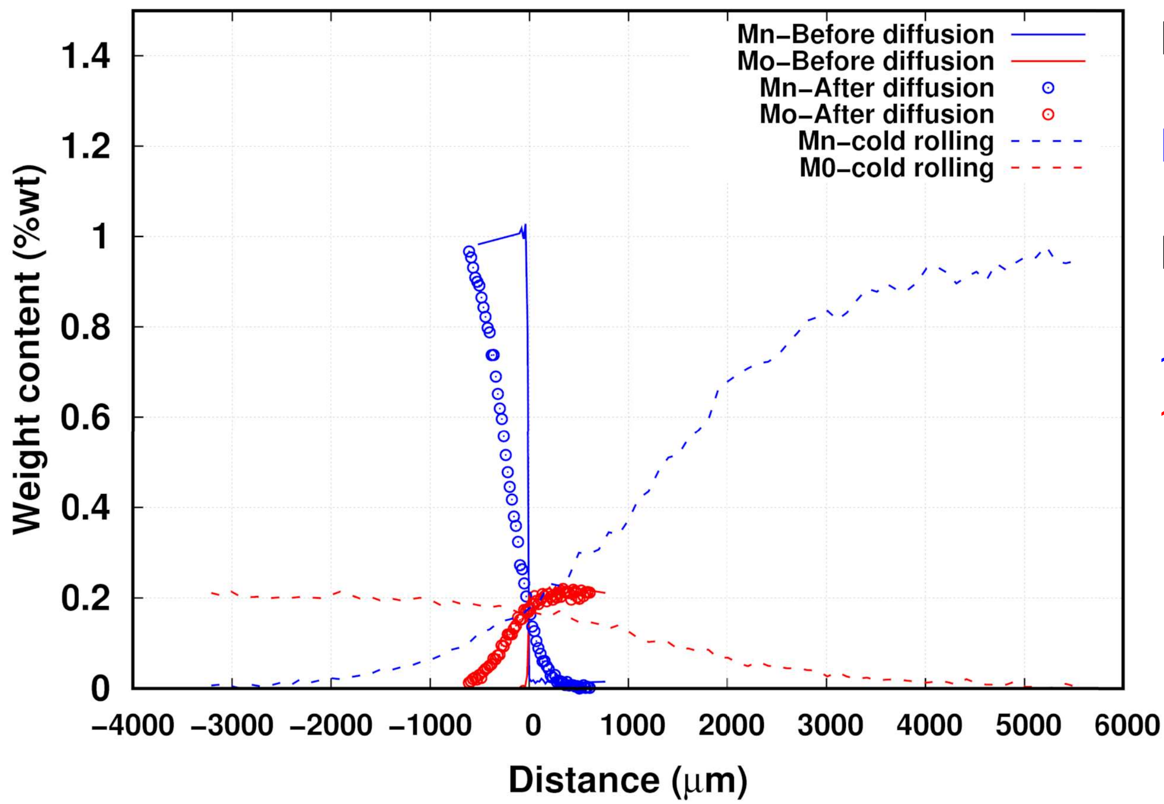
950µm (Mn profile)
700µm (Mo profile)

# Methodology of CGM fabrication

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

3 Extending by cold/hot rolling

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 cold rolling

~7 mm (Mn profile)
~4.5mm (Mo profile)

# What’s next ?

# What's next ?

- 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 – October - November 2018
ESRF ( beamline ID11, HEXRD 100keV, beam size 200-400µm)

# What's next ?

- 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 – October - November 2018
ESRF ( beamline ID11, HEXRD 100keV, beam size 200-400µm)

Procedure : Austenitization 900°C – 10min
Quenching to phase transformation temperature (700°C to 850°C)
Isothermal holding : up to 1 hour
Quench to room temperature

# What's next ?

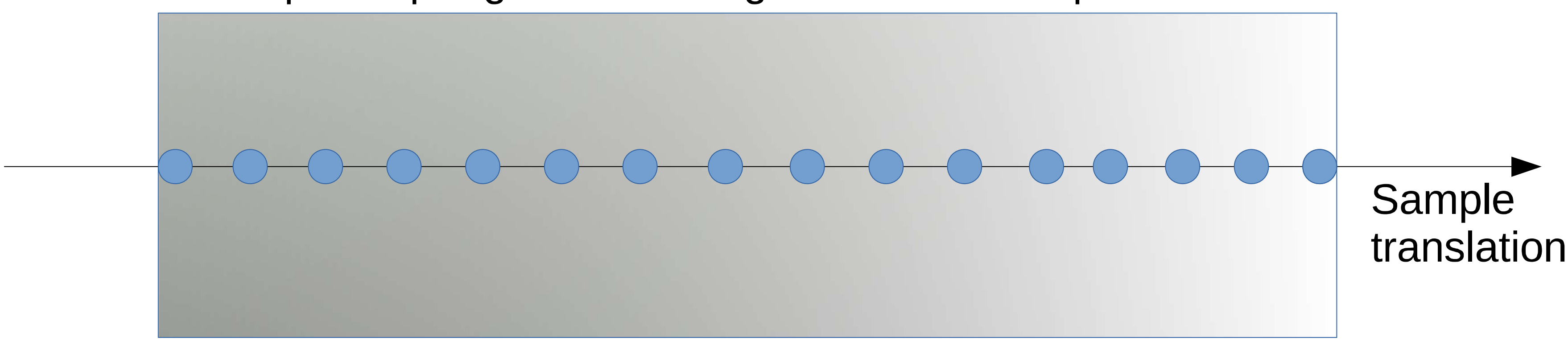
- Implementing a new methodology for the fabrication of **compositionally-graded materials.**

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

Synchrotron beam time : 12 shifts – October - November 2018

ESRF ( beamline ID11, HEXRD 100keV, beam size 200µm)

20 points per gradient – 10 gradients – 4 temperatures

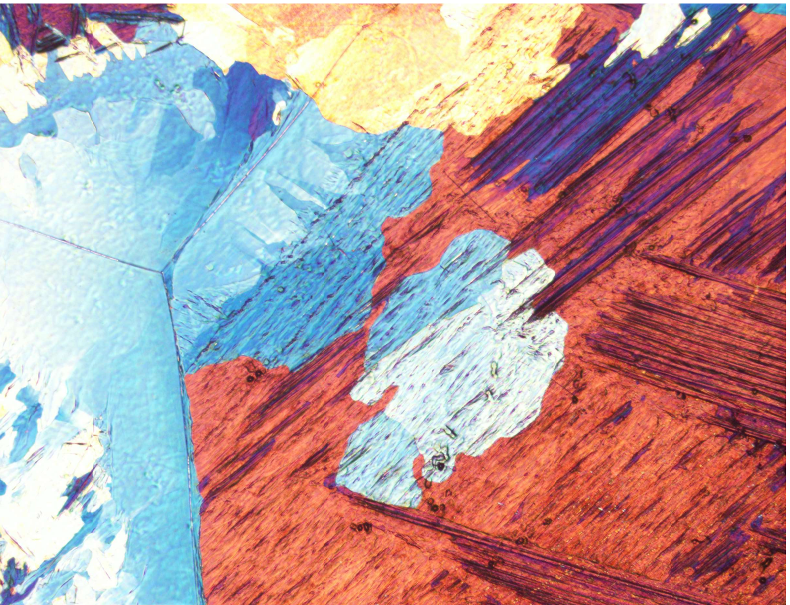


# What's next ?

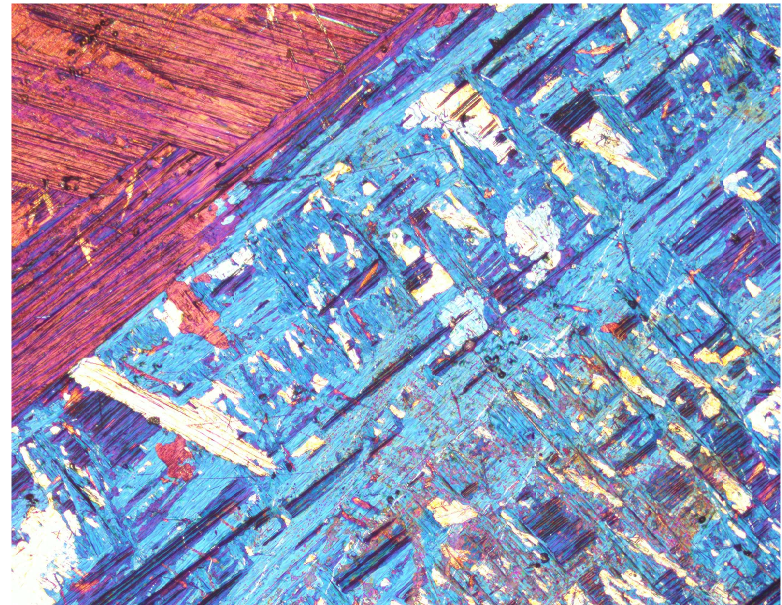
- Implementing a new methodology for the fabrication of **compositionally-graded materials.**

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

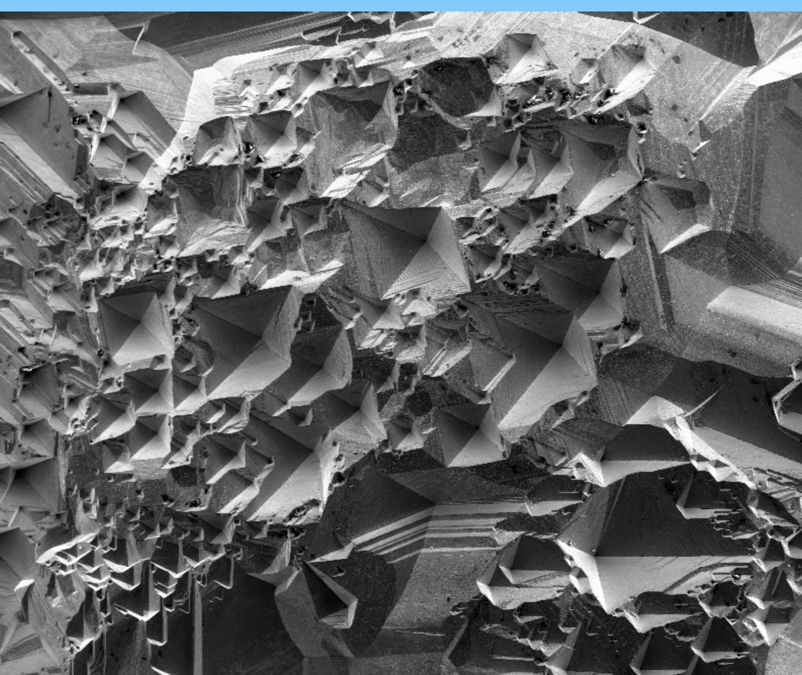
- APT (atom probe tomography) and TEM (transmission electron microscopy) techniques will be used on the treated samples to investigate the segregation at the migrating interface.



Decarburized



# Thank You



Carburized