

# Development of an in-situ tensile test at high temperature within a SEM in order to quantify the plastic strain of the AZ31 Mg-alloy

05/06/2018

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Advisors : Martin Guilhem and Lhuissier Pierre

Collaboration : Delannay Laurent



**1. Context**

**2. Development of an in-situ tensile test at high temperature within a SEM**

**3. Example: plastic strain heterogeneities at 250°C**

**4. Outlook**

# Industrial application of Mg-alloys and forming

- ➔ Automotive
- ➔ Aeronautics
- ➔ Free casting
- ➔ Wrought material :  
rolling, extrusion, ...



[Credits General Motor]



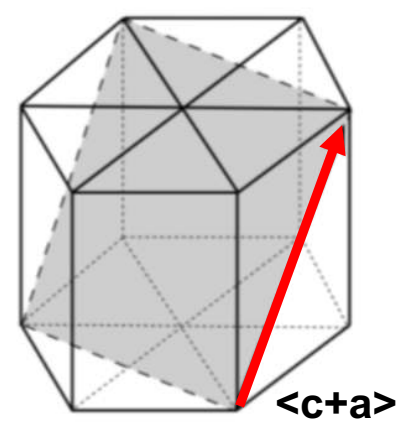
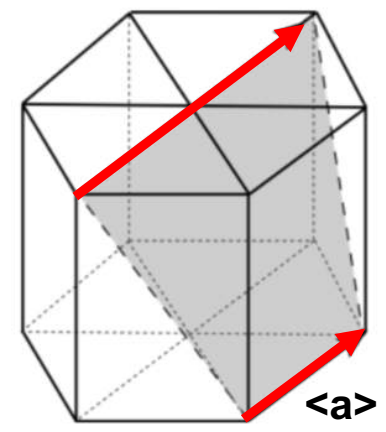
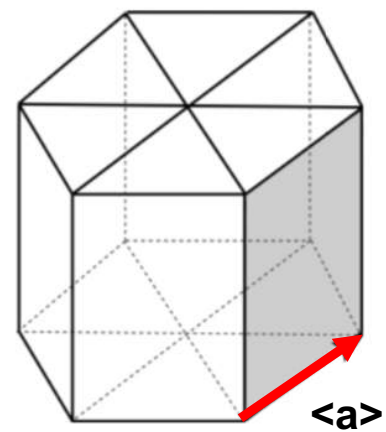
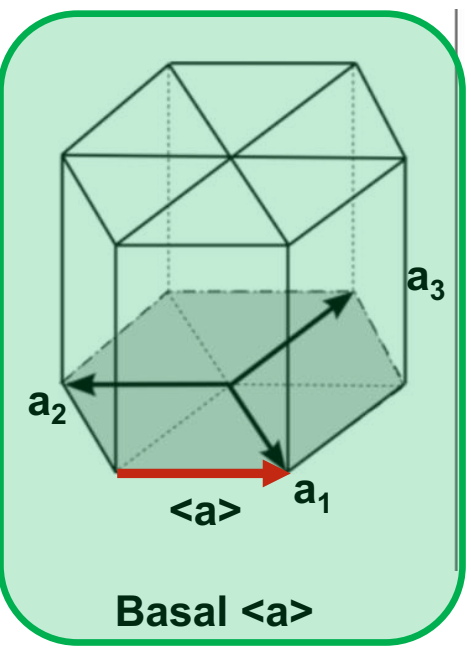
Properties	Magnesium	Aluminium 7050
Density (g/cm <sup>3</sup> )	1,74	2,83
Young modulus (GPa)	44	72
Specific Young modulus (GPa.g <sup>-1</sup> .cm <sup>-3</sup> )	25,28	25,44

[Lide, Handbook of Chemistry & Phy. (2003)]

➔ **Interesting specific mechanical properties, forming by plastic deformation at RT is challenging**

# Deformation mechanisms at RT

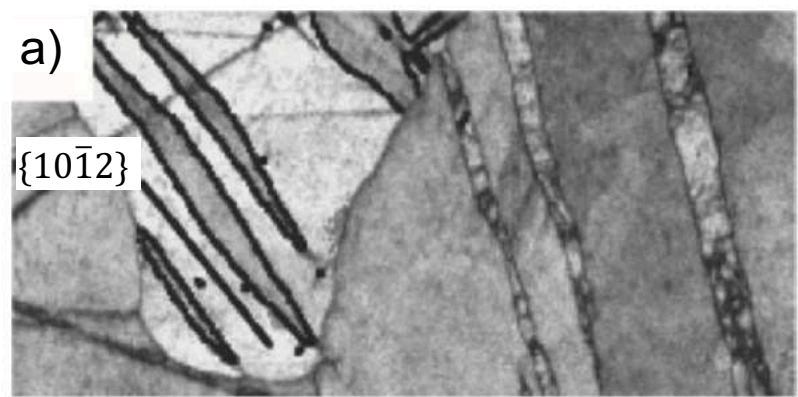
⇒ Dislocation glide



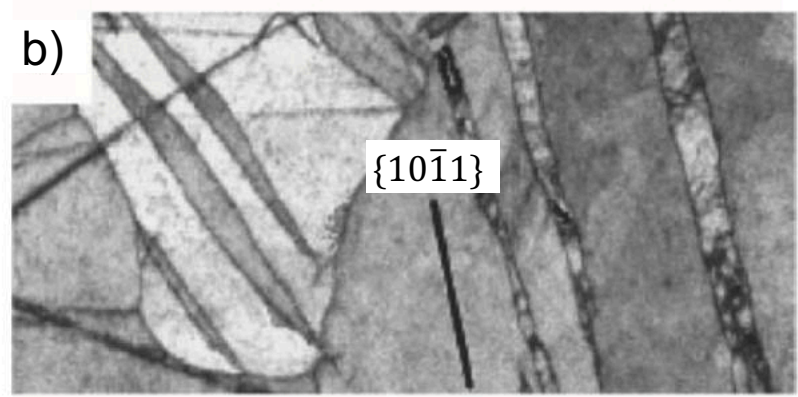
Deformation is controlled by basal dislocation

# Deformation mechanisms at RT

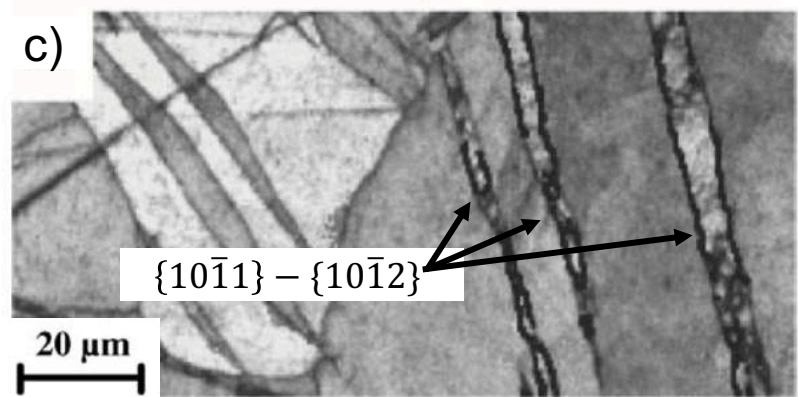
⇒ **Twinning**



➔ **Tensile twin**



➔ **Compression twin**

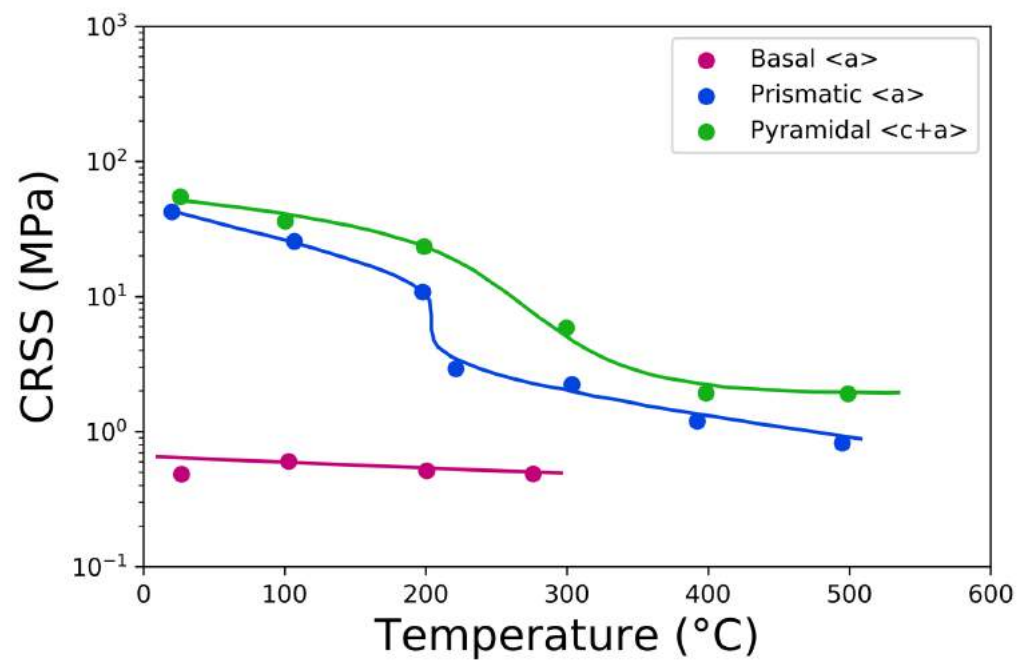


➔ **Double twin**

[Nave and Barnett, Scripta Materialia, (2004)]

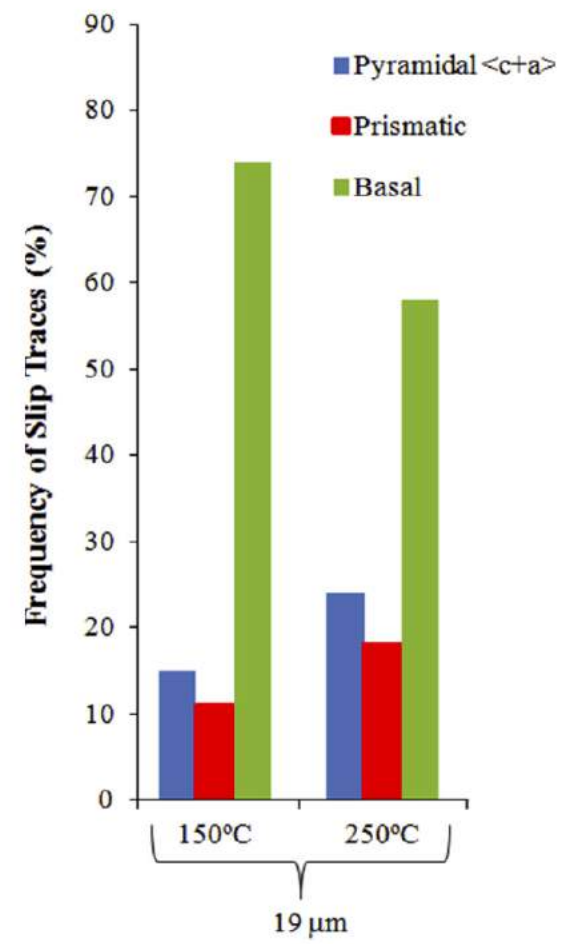
# Deformation mechanisms at HT

⇒ Dislocation creep



[Barnett, Metallurgical and Materials Transactions, (2003)]

➔ With temperature => activation of No-basal slip become more easier



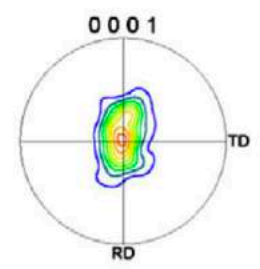
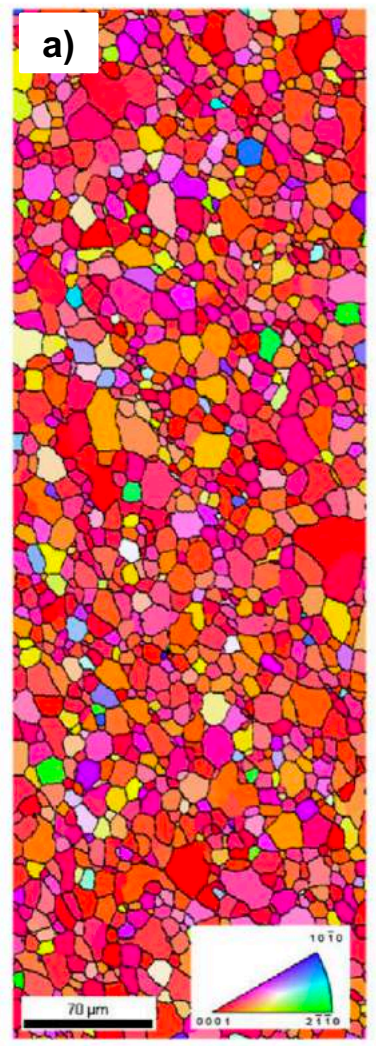
[Cepeda-Jimenez et al., Acta Materialia, (2015)]

# Deformation mechanisms at HT

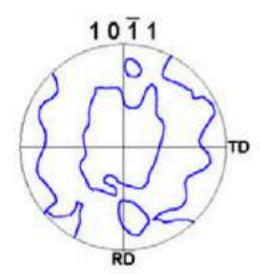
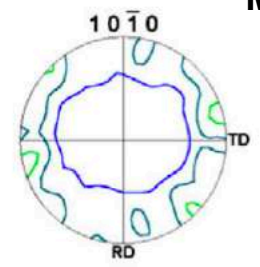
⇒ Recrystallization



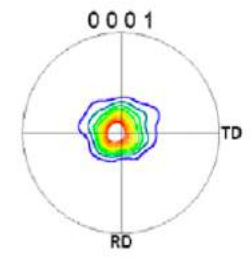
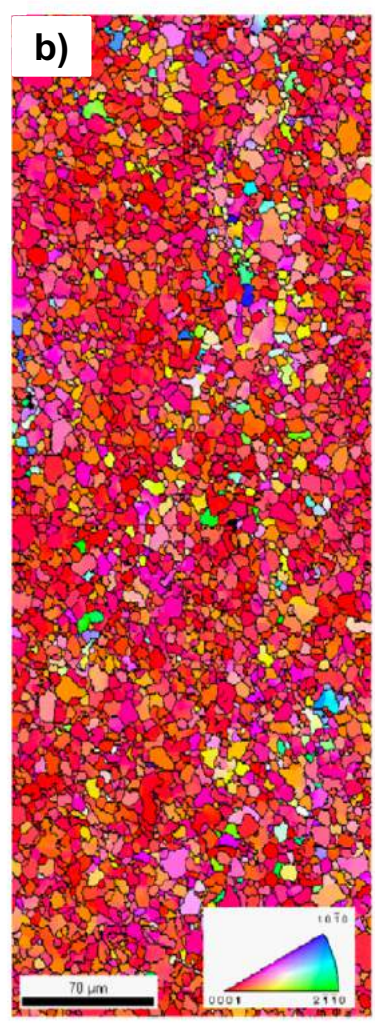
Recrystallization function to temperature and strain rate



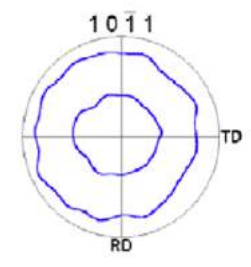
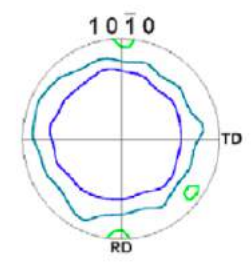
Max = 8.5



Grain size = 15µm



Max = 9

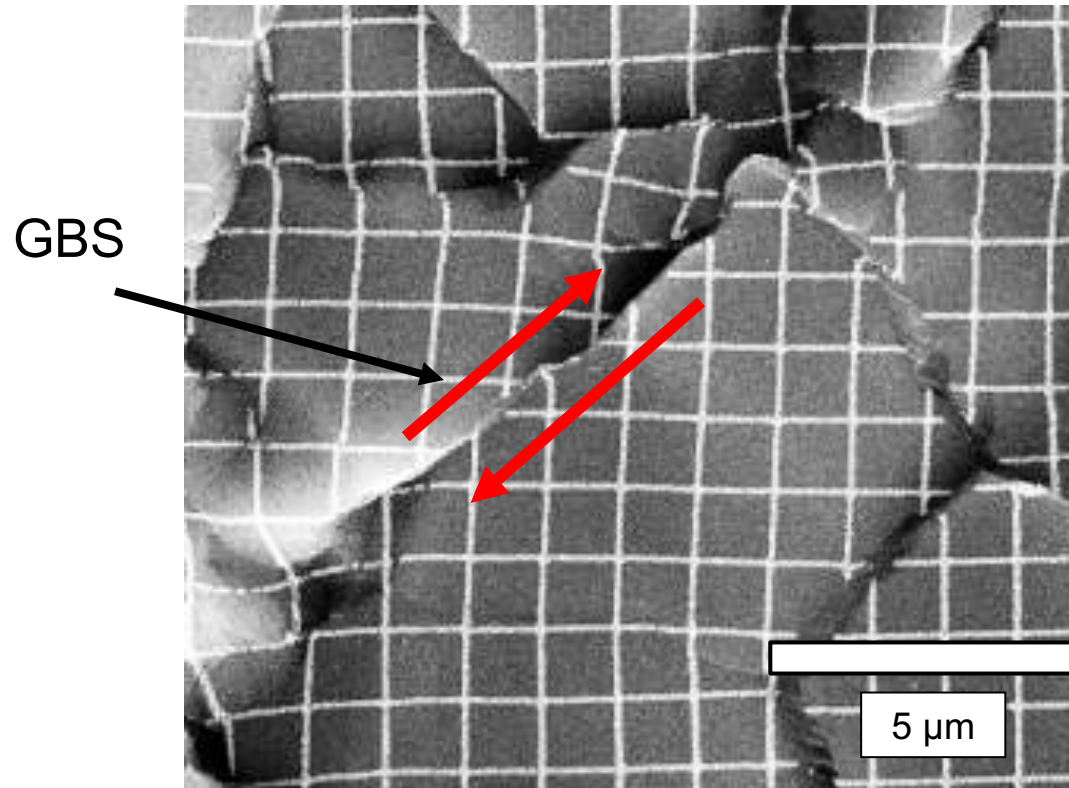


Grain size = 7µm

[Ulacia et al., Acta Materialia, (2010)]

## Deformation mechanisms at HT

⇒ **GBS**



*[SIMaP, Unpublished work (2017)]*

➡ **Few data on in-situ GBS on Mg alloy in literature**

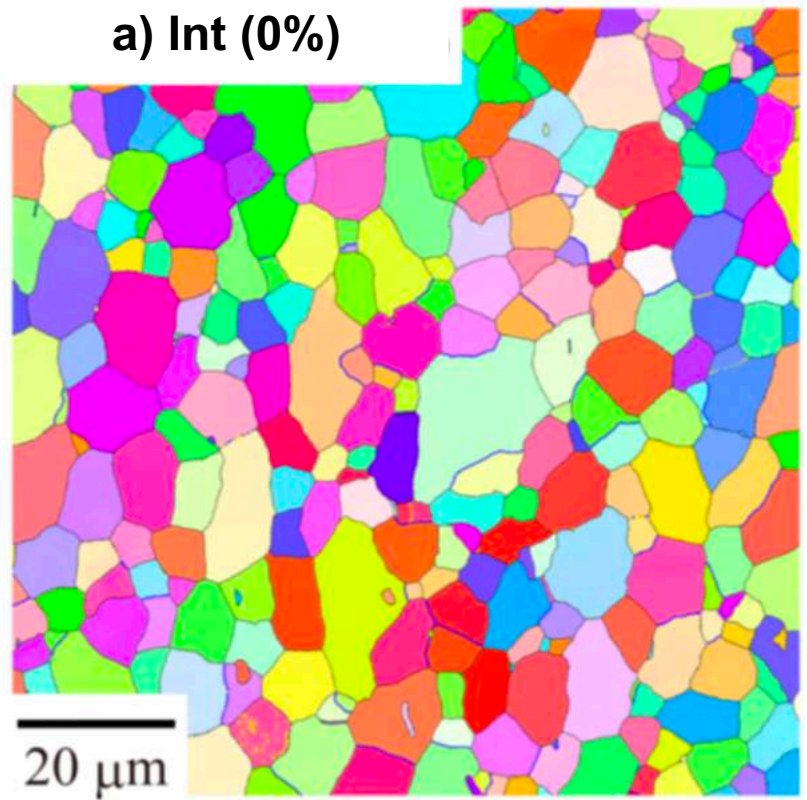
➡ **When grain size is smaller and temperature important**

# Deformation mechanisms at HT

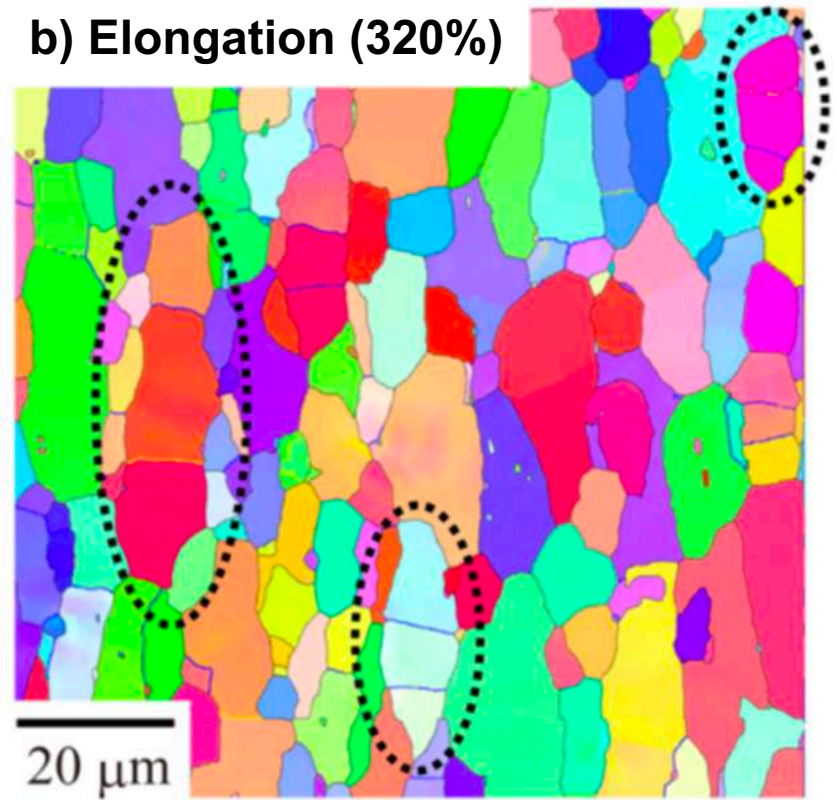
⇒ Diffusion and grain growth

[Masuda et al., Scripta Materialia , (2018)]

a) Int (0%)



b) Elongation (320%)



➡ Grain size : 4μm to 13μm

➡ Diffusion in lattice or grain boundaries

➡ High temperature and small grain size

# Deformation mechanisms at HT

- ⇒ **Dislocation creep**
- ⇒ **Recrystallization**
- ⇒ **GBS**
- ⇒ **Diffusion and grain growth**



**Function to :**

- **Material**
- **Temperature**
- **Strain rate**
- **Grain size**



**Coexistence of deformation mechanisms at HT**

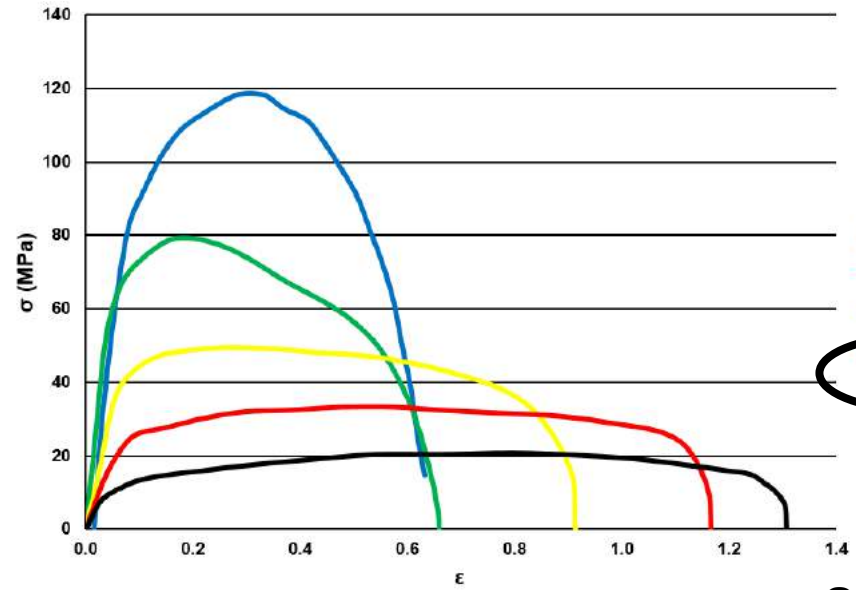
# Deformation mechanisms at HT

⇒ Superplasticity

→ Requires specific conditions to be activated

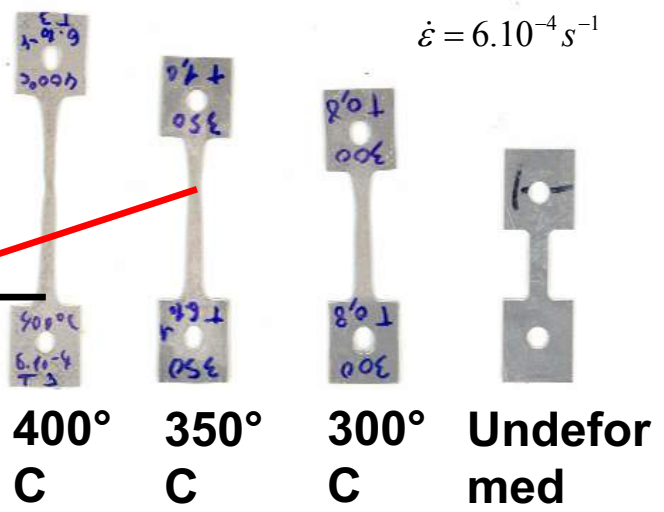
- Small grains
- Slow and controlled strain rate
- Restricted temperature windows

Effect of temperature on tensile curve for AZ31 Mg alloy at  $\dot{\epsilon} = 6.10^{-4} s^{-1}$



350°C

Superplasticity



[R. Boissière, PhD Thesis, SIMaP/GPM2 (2008)]

## Deformation mechanisms vs. Temperature

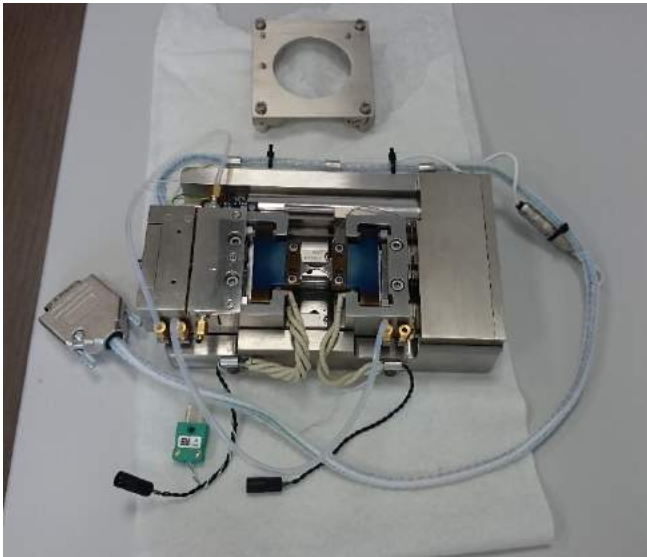
		Room Temperature	HT plasticity	Superplasticity
<b>Dislocation glide</b>	<i>Basal slip</i>	+++	?	?
	<i>Prismatic slip</i>	+	?	?
	<i>Pyramidal &lt;a&gt; slip</i>	→0	?	?
	<i>Pyramidal &lt;c+a&gt; slip</i>	→0	?	?
<b>Twinning</b>				
	<i>Tensile twinning</i>	+++	→0	→0
	<i>Compression twinning</i>	+	→0	→0
<b>Grain boundary sliding</b>		→0	+	+++
<b>Diffusion</b>		→0	+	+++

Understanding the transition

→ Objective : Quantifying the deformation mechanisms operating during high temperature to superplastic deformation of the AZ31 Mg-alloy: from 200 to 350°C.

# Development of an in-situ tensile test at high temperature within a SEM

## In-situ tensile stage



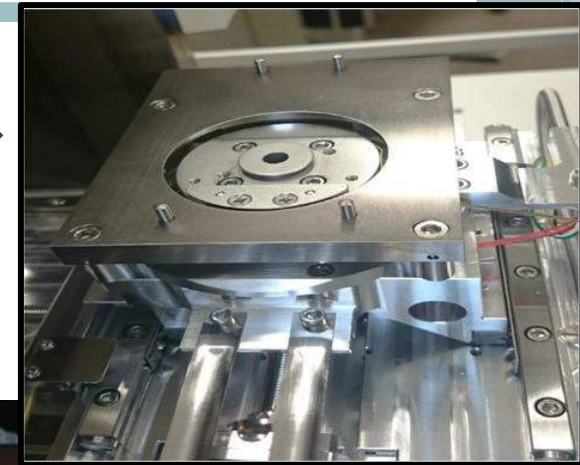
**Deben/Gatan In-Situ Tensile Stage**



**SEM ZEISS GEMINI 500**

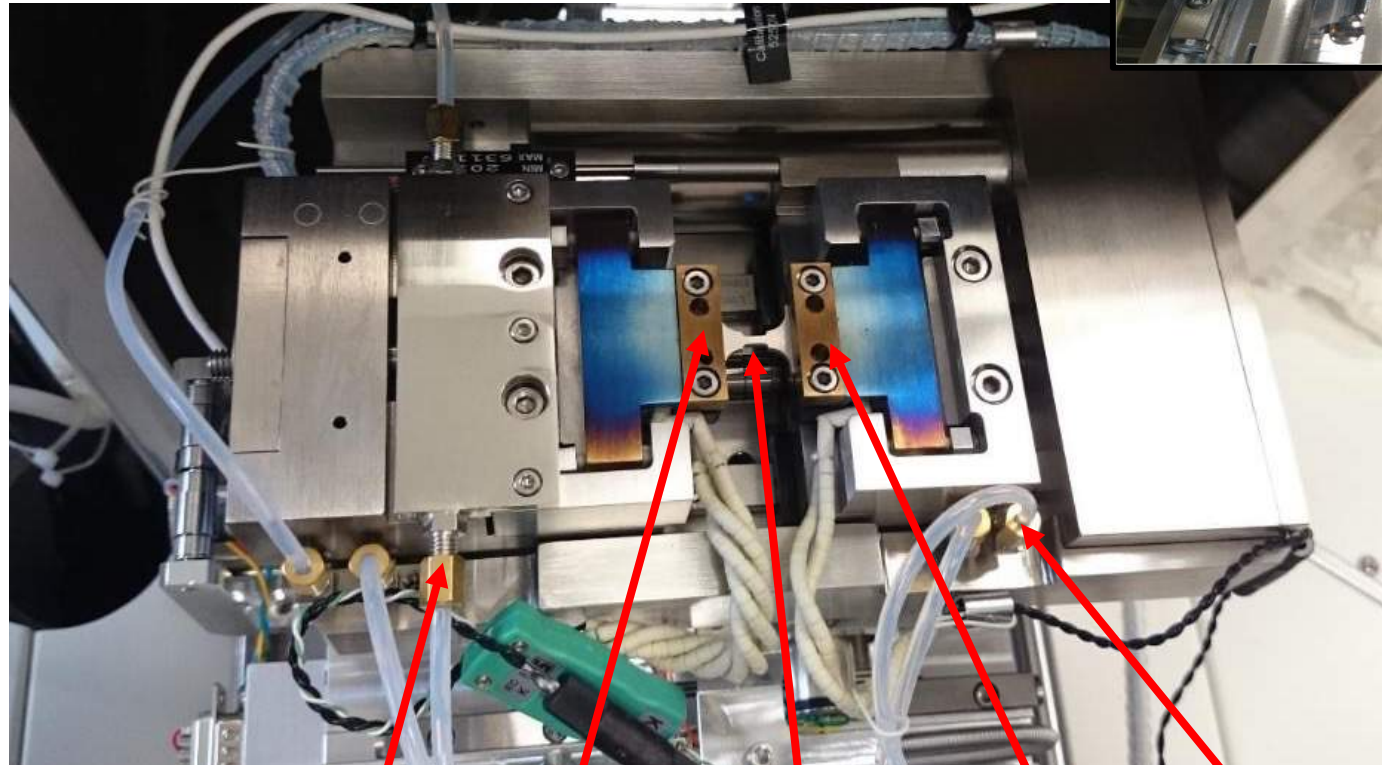
## In-situ tensile stage

Adaptation  
piece →



### Gatan MT5000W stage :

- ⇒ Cell force : 5kN or **200N**
- ⇒ Heated flat jaw
- ⇒ Range of strain rate :  $3 \cdot 10^{-5} \text{ (s}^{-1}\text{)} - 7 \cdot 10^{-3} \text{ (s}^{-1}\text{)}$



Cooling  
system

Hot jaws

Sample

Hot jaws

Cooling  
system

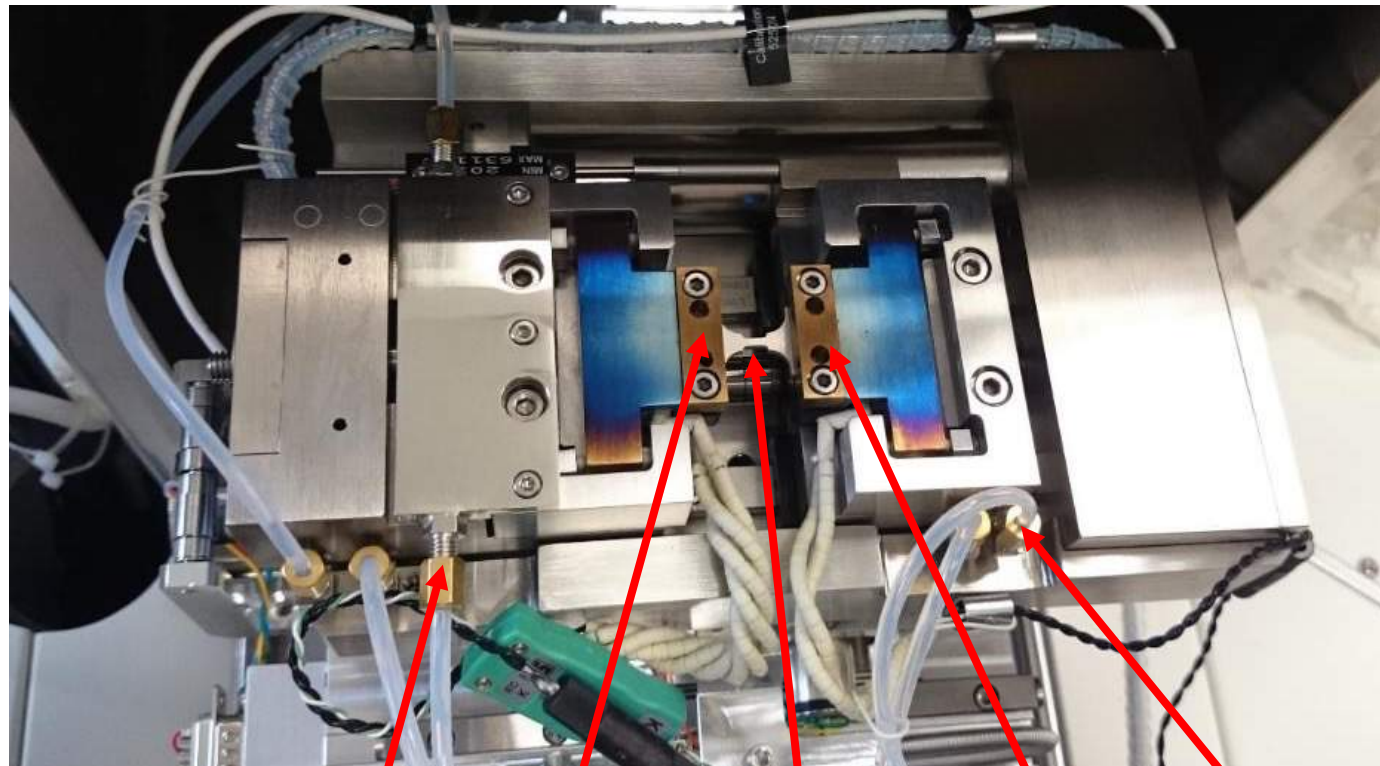
## In-situ tensile stage

Control of strain rate by  
a « home-made »  
Labview program

### Gatan MT5000W stage :

- ⇒ Cell force : 5kN or **200N**
- ⇒ Heated flat jaw
- ⇒ Range of strain rate :  $3 \cdot 10^{-5} \text{ (s}^{-1}\text{)} - 7 \cdot 10^{-3} \text{ (s}^{-1}\text{)}$

Fix WD



Cooling  
system

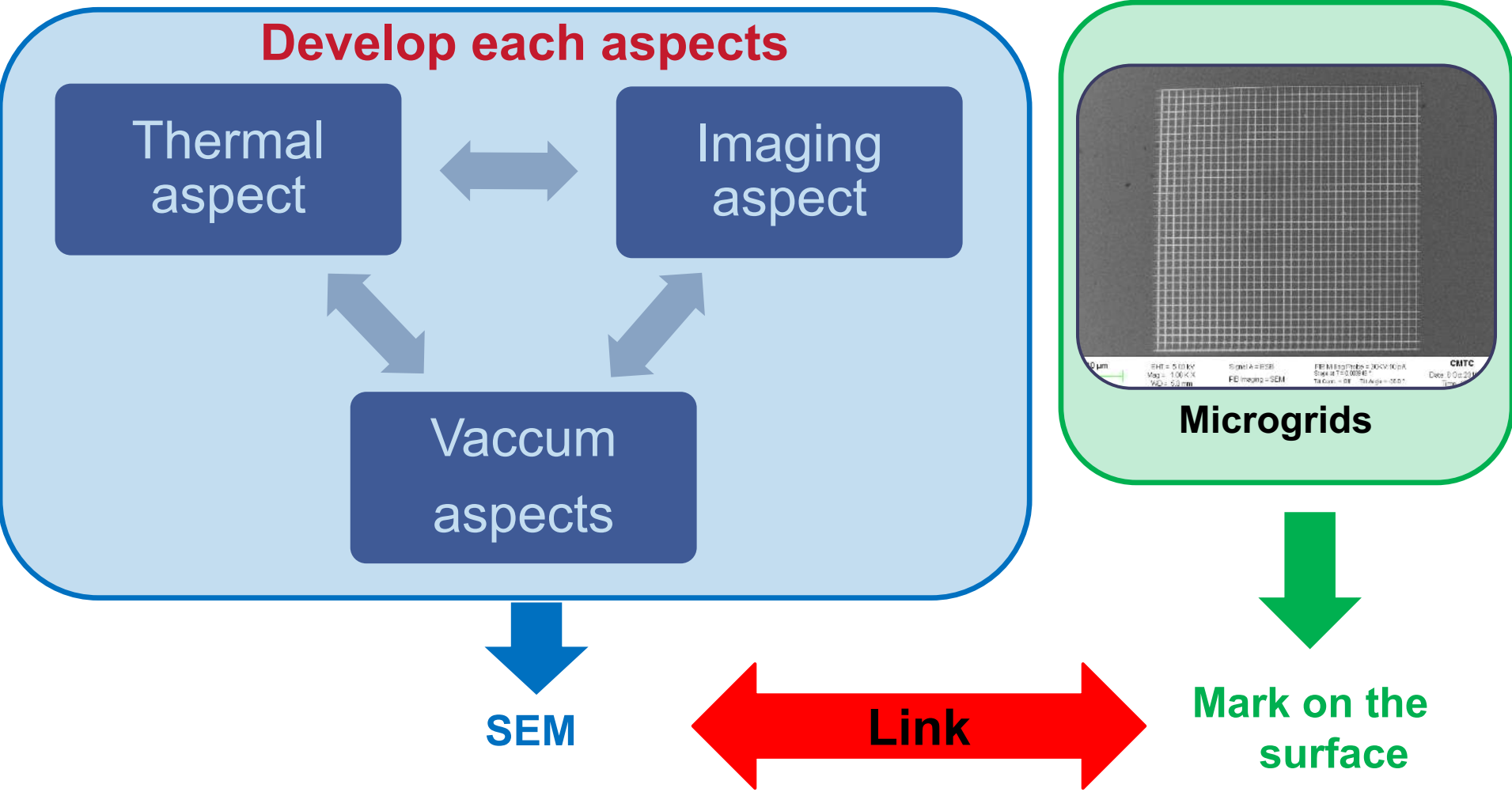
Hot jaws

Sample

Hot jaws

Cooling  
system

# Development of in-situ tensile test





## Development of in-situ tensile test

⇒ Microgrid on the surface

AZ 31 : single-phased → **No local contrast for Digital Image Correlation (DIC) in the microstructure**

Requirement: add markers on the surface

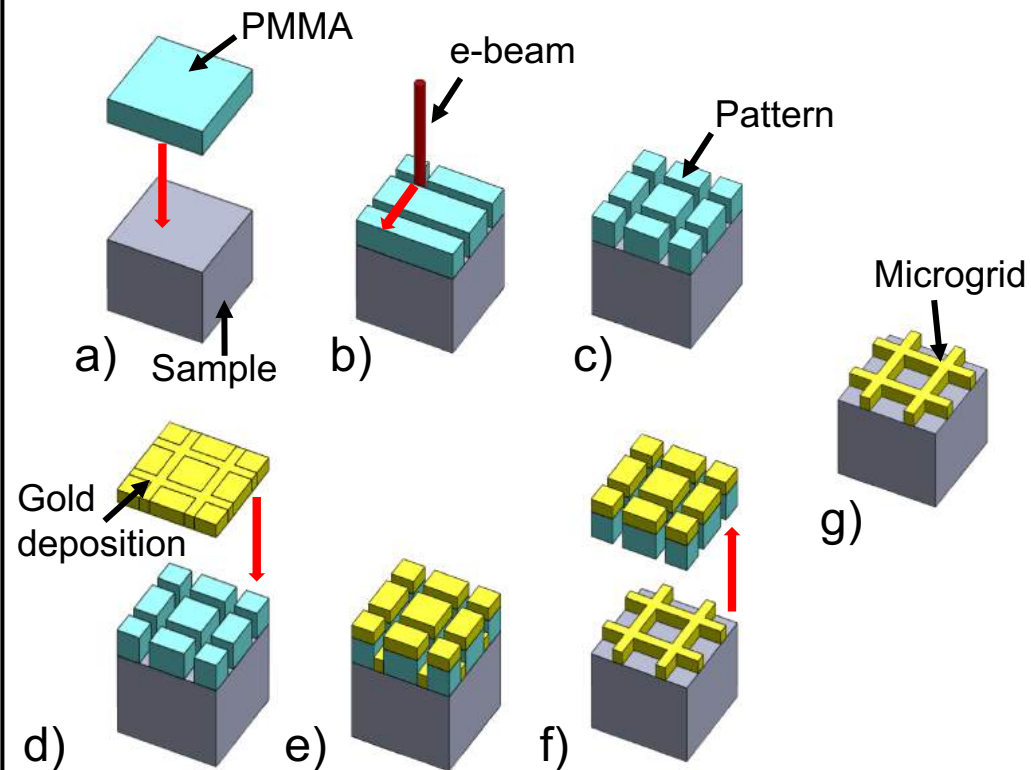


**Microgrids made by E-beam lithography**

⇒ Microgrids enable to highlight the GBS mechanism

⇒ Repetable process

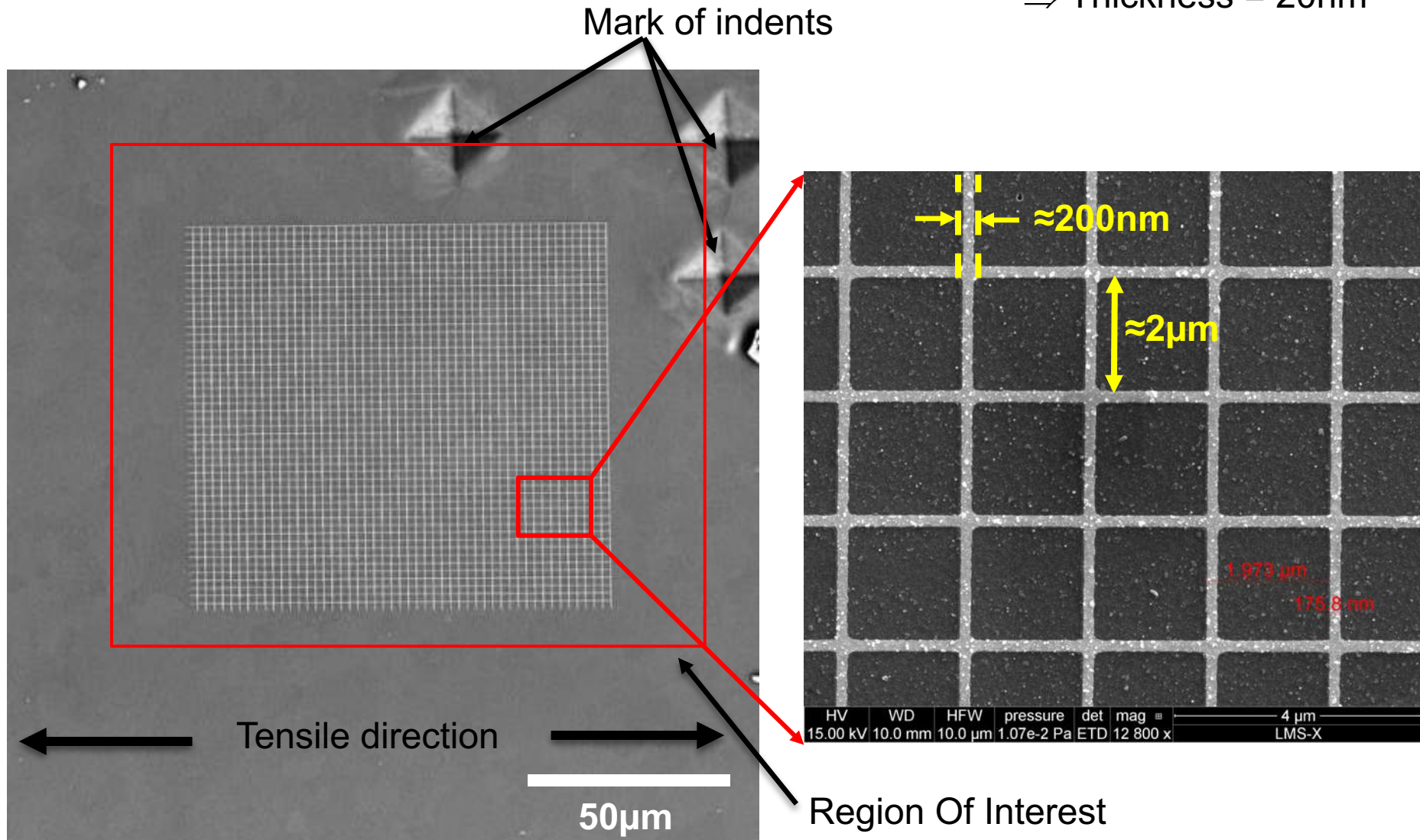
### E-beam lithography :



## Development of in-situ tensile test

⇒ **Microgrid on the surface**

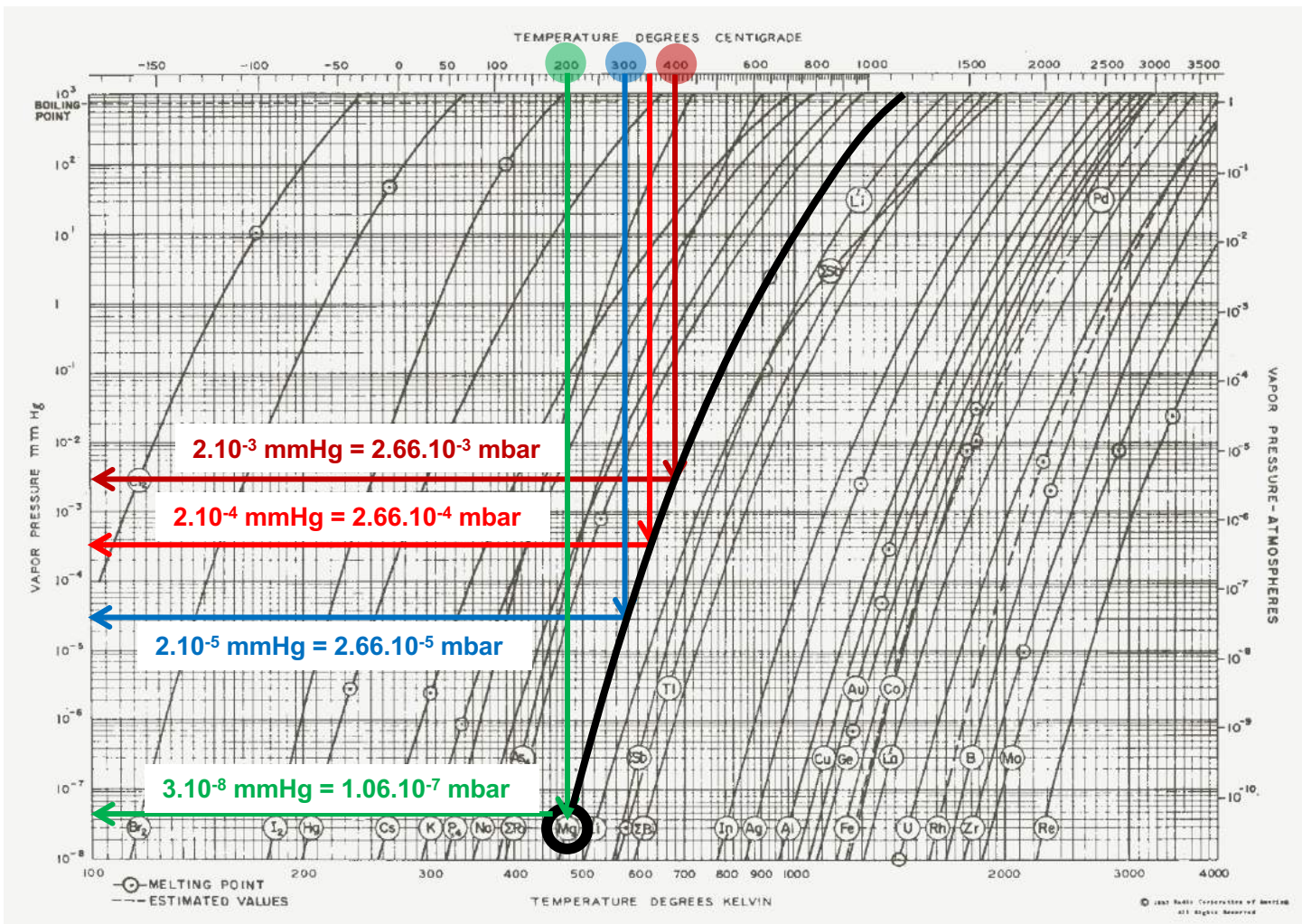
- ⇒ Size : 100x100 $\mu$ m
- ⇒ Material : Gold
- ⇒ Thickness = 20nm



# Development of in-situ tensile test

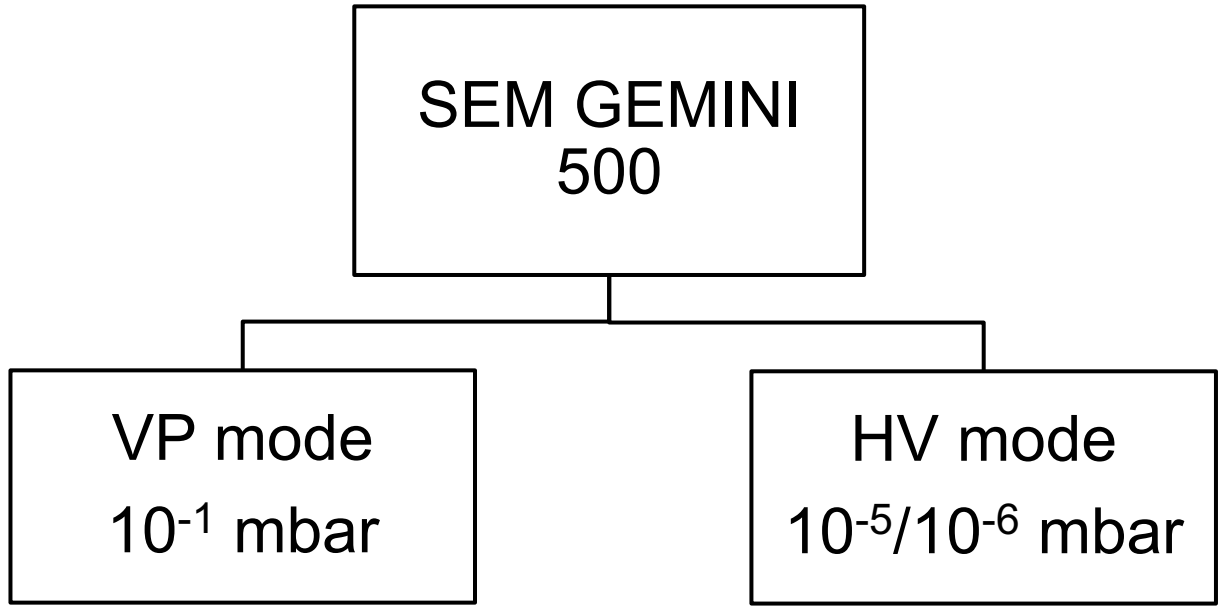
⇒ Vacuum aspect

## IMPORTANT REQUIREMENT : sublimation risk



# Development of in-situ tensile test

⇒ Vacuum aspect

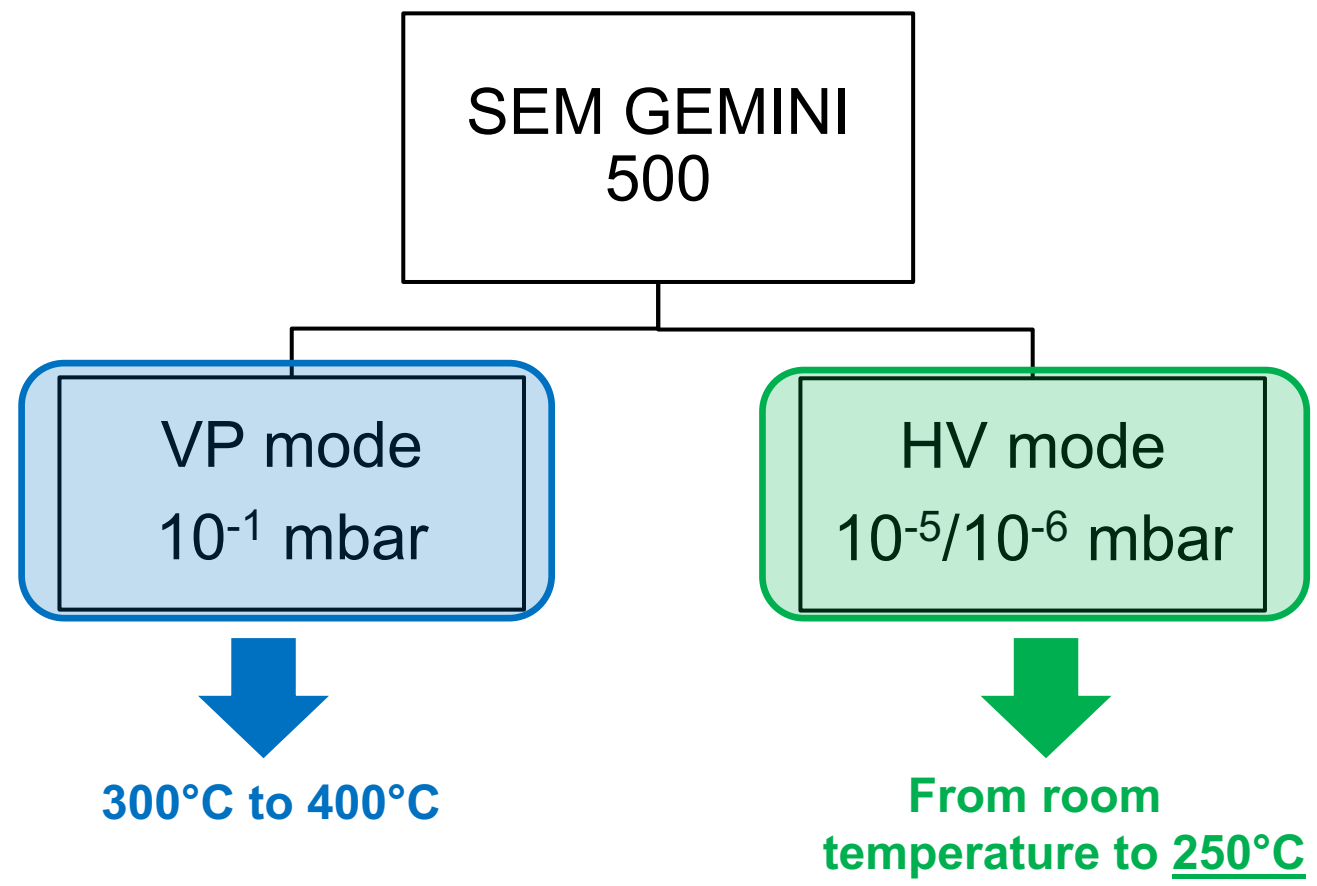


Temperature list :

$400^{\circ}\text{C} = 2.66 \cdot 10^{-3} \text{ mbar}$	}	Obligation of VP mode
$350^{\circ}\text{C} = 2.66 \cdot 10^{-4} \text{ mbar}$		
$300^{\circ}\text{C} = 2.66 \cdot 10^{-5} \text{ mbar}$	}	Potential risk to work in HV mode
$200^{\circ}\text{C} = 1.06 \cdot 10^{-7} \text{ mbar}$	}	No problem

# Development of in-situ tensile test

⇒ Vacuum aspect



## Development of in-situ tensile test

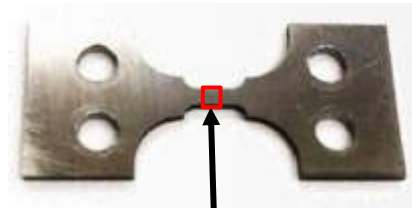
⇒ Thermal aspect

2 aspects :

↳ Sample

↳ Pieces inside the chamber of SEM

➔ Sample



Spot welded thermocouple

↳ Verification of temperature in the jaw

↳ Gap inferior of 10°C

## Development of in-situ tensile test

⇒ Thermal aspect

2 aspects :

↳ Sample

↳ Pieces inside the chamber of SEM

---

↳ Pieces inside the chamber of SEM

↳ In VP mode

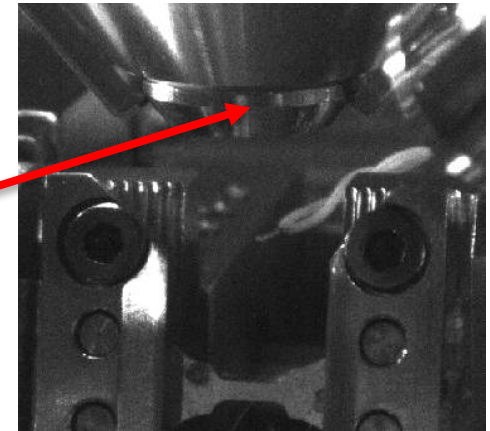
**$T_{\max}$  for lens = 100°C**

↳ Verification of temperature at different areas

➔ **Installation of a thermal shield**

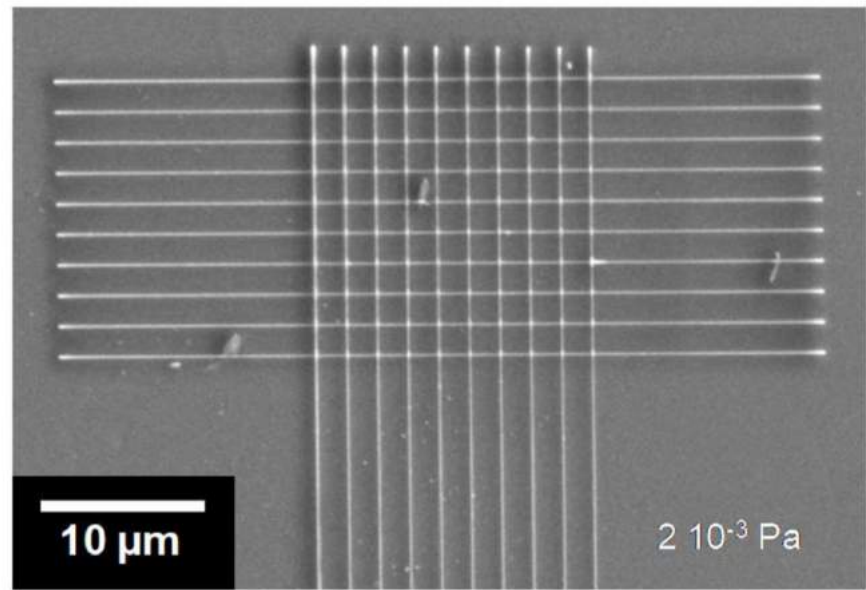
➔  **$T_{\max}$  = 300°C**

Polar part of the SEM

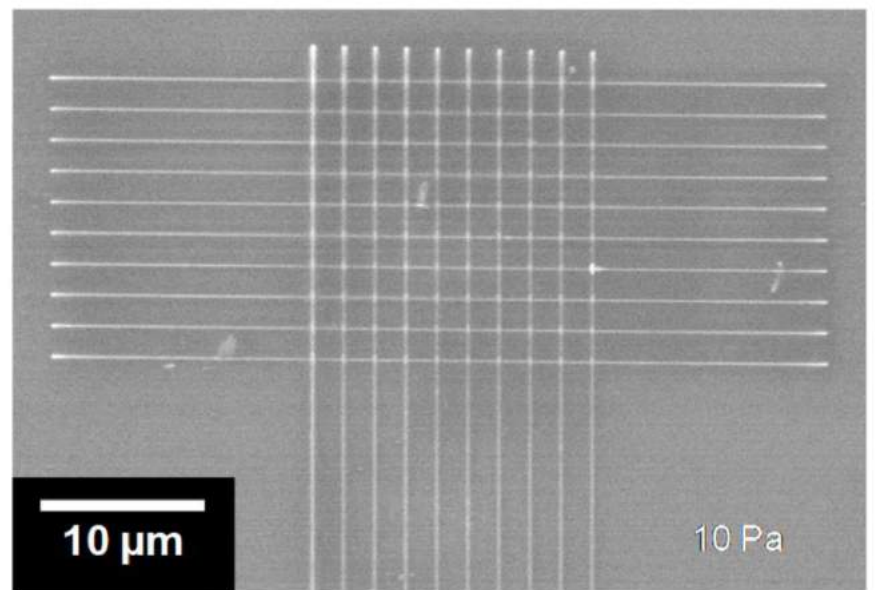


# Development of in-situ tensile test

⇒ Imaging aspect



**HV mode**  
**All detector available**



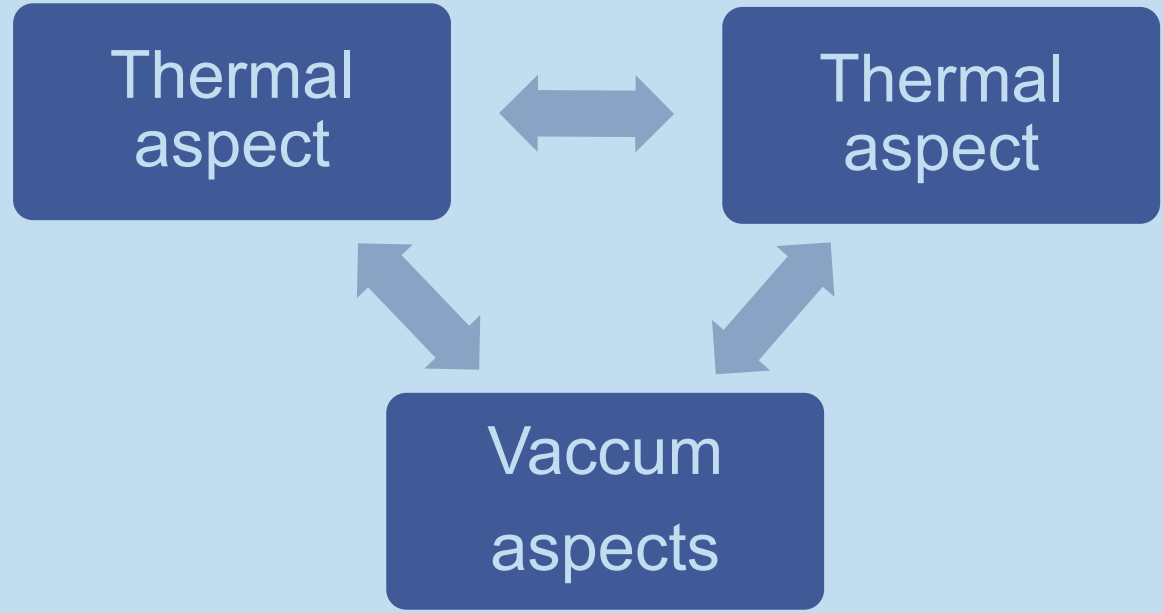
**VP mode**  
**VPSE detector**

➔ Thermal shield

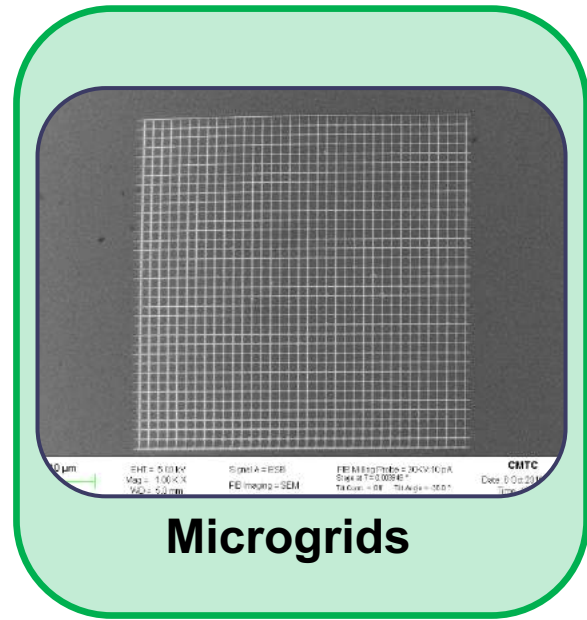
➔ Optimisation imaging for DIC

# Development of in-situ tensile test

## Develop each aspects



SEM



Mark on the surface

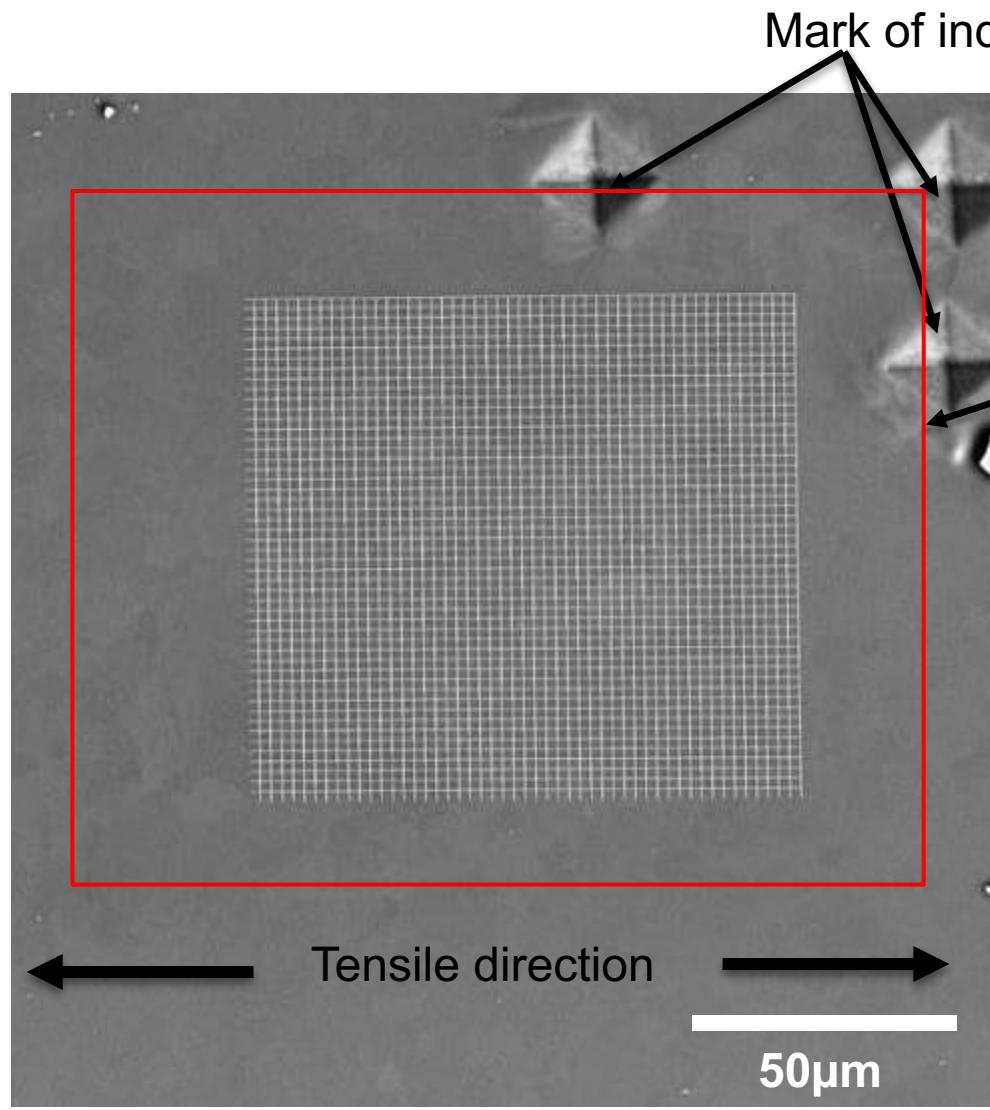
Link

Control each parameters

# Plastic strain heterogeneities at 250°C

# EBSD before the e-beam lithography

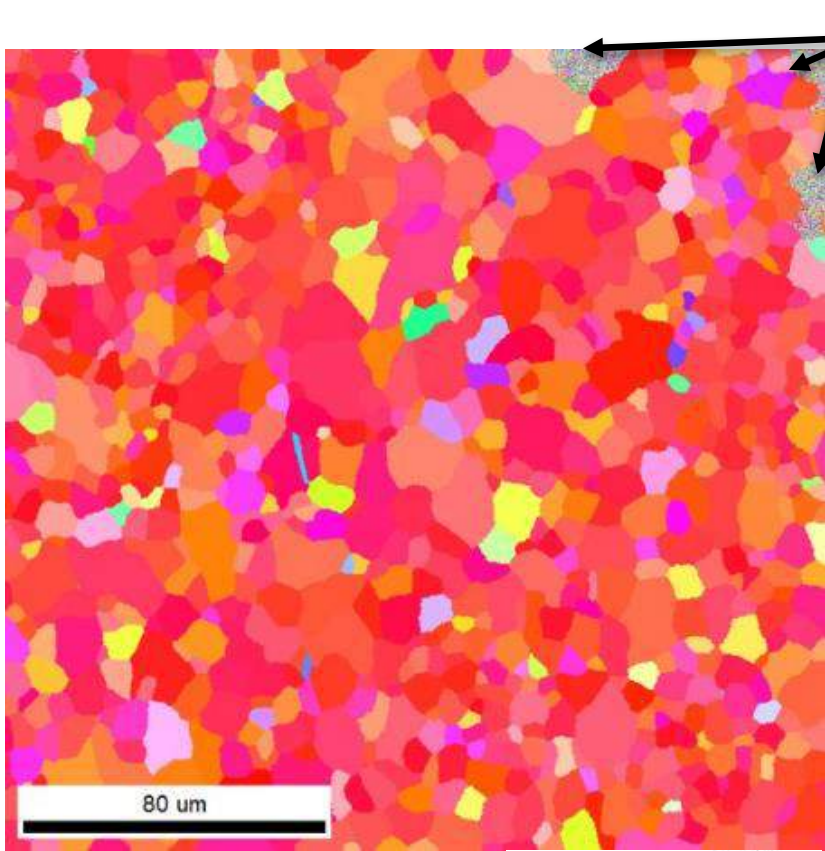
- ⇒ Size : 100x100μm
- ⇒ Material : Gold
- ⇒ Thickness = 20nm



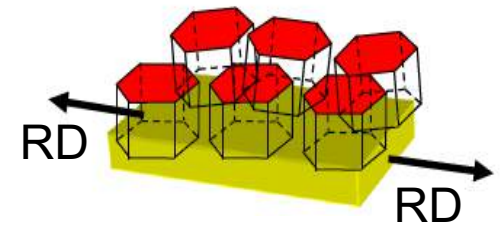
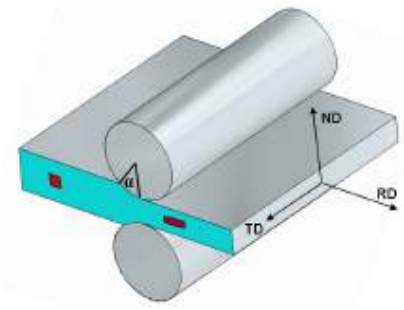
EBSD mapping of this region of interest before e-beam lithography

- ➔ Crystallographic informations
- ➔ Grains boundaries

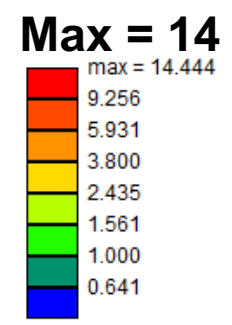
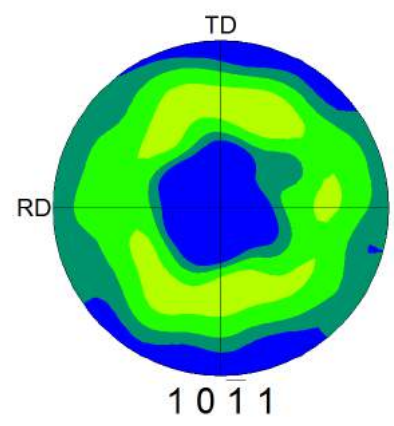
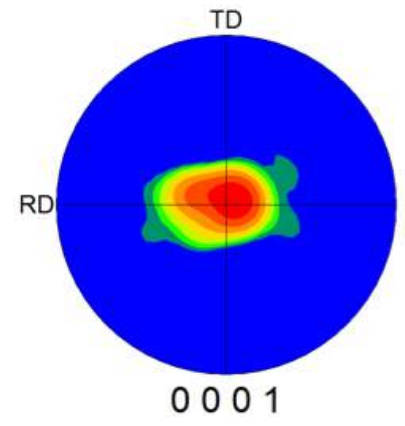
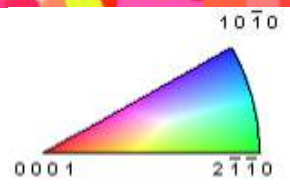
# Initial microstructure of AZ31 Mg alloy



Mark of indents

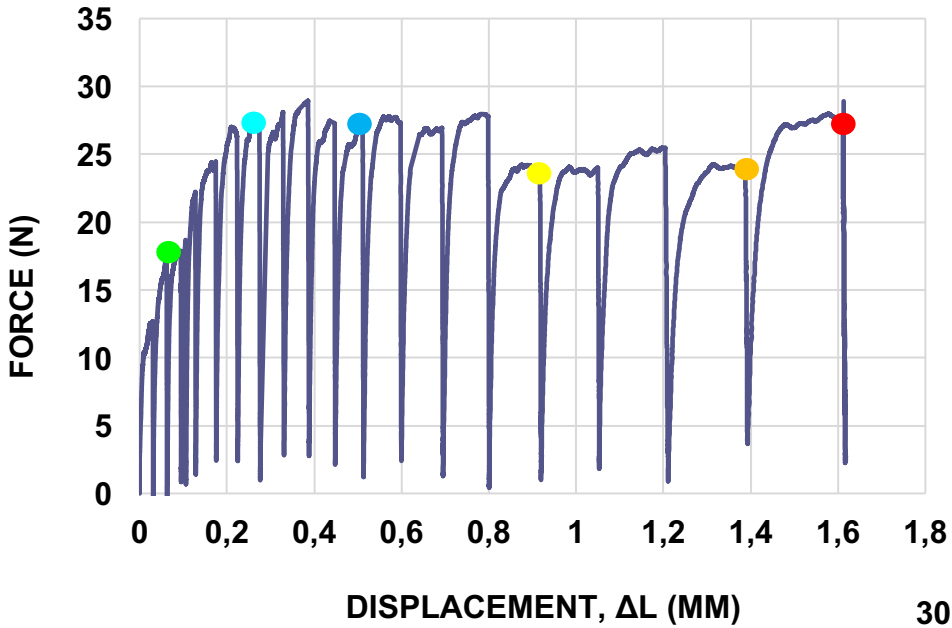


IPF-EBSD map

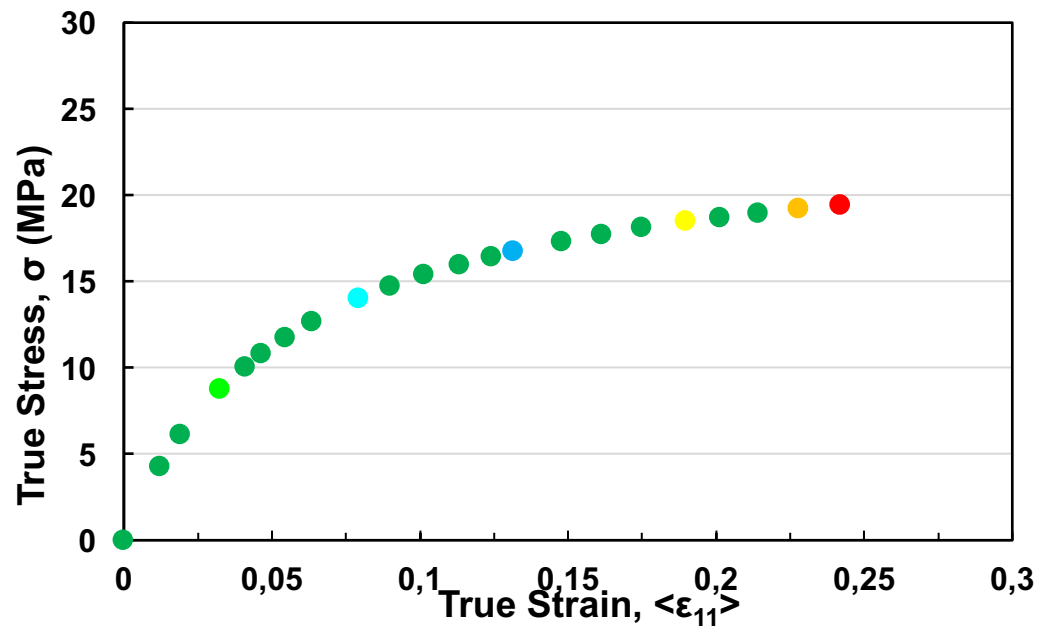


Average grain size  $\approx 12\mu\text{m}$

# Mechanical behavior

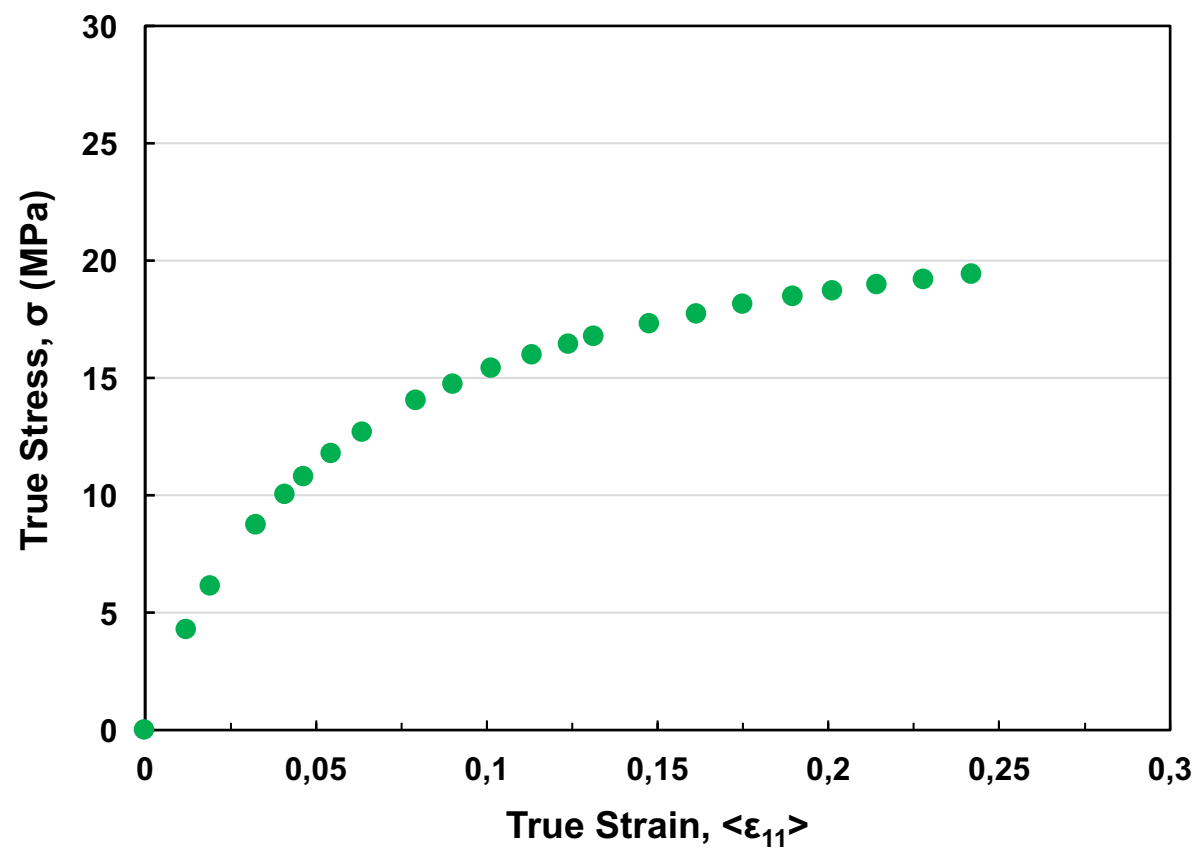


➔ Strain Rate =  $5 \cdot 10^{-5} \text{ s}^{-1}$   
T = 250°C

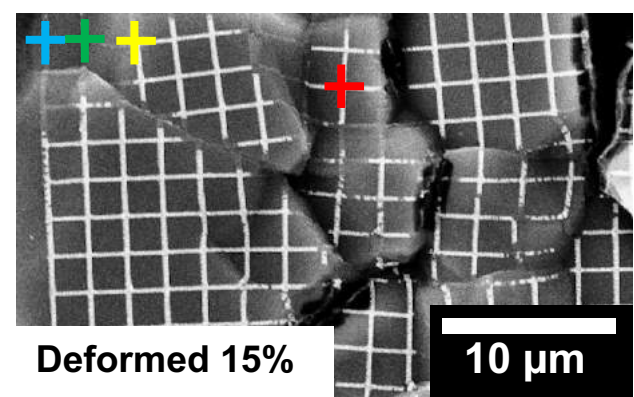
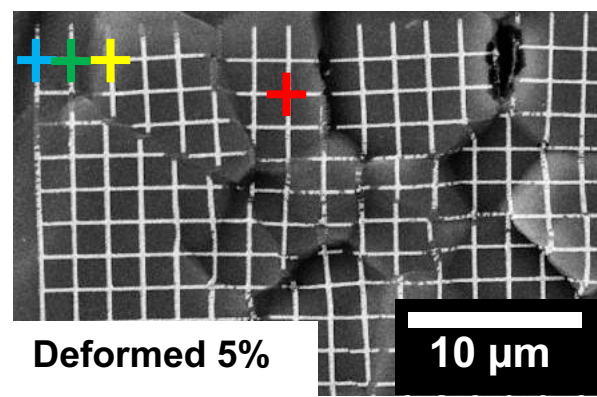
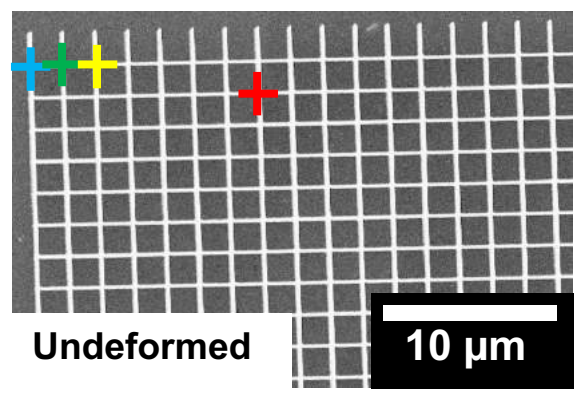


# Mechanical behavior

➔ Strain Rate =  $5 \cdot 10^{-5} \text{ s}^{-1}$   
T = 250°C



# Strain map

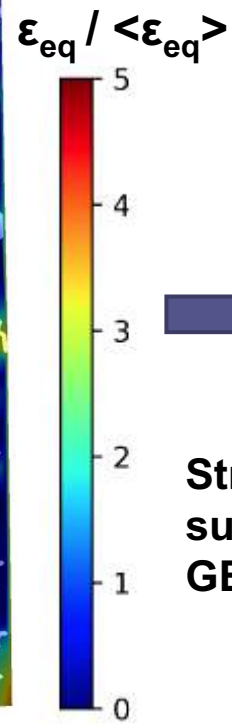
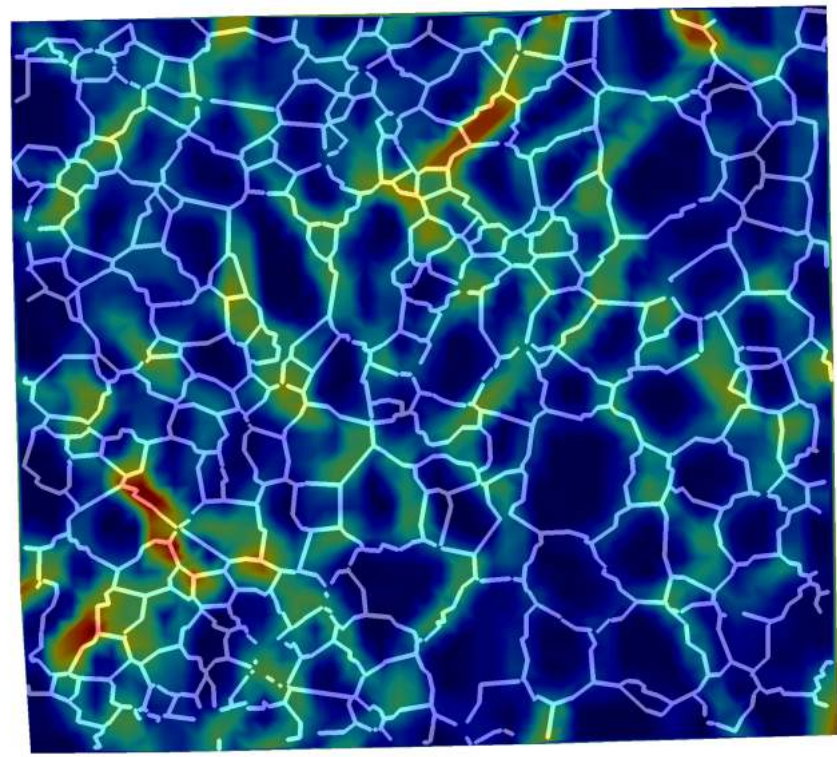


20 μm

250°C

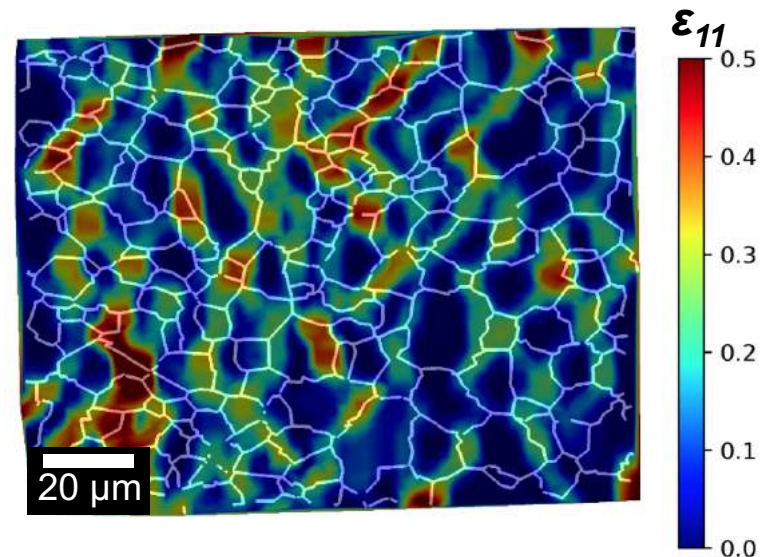
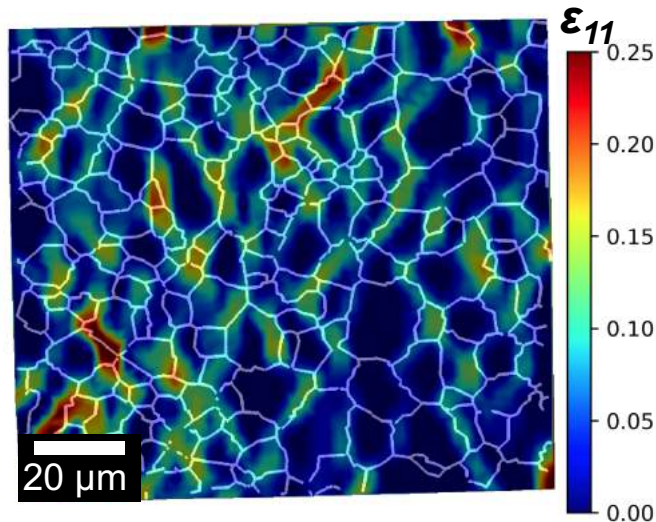
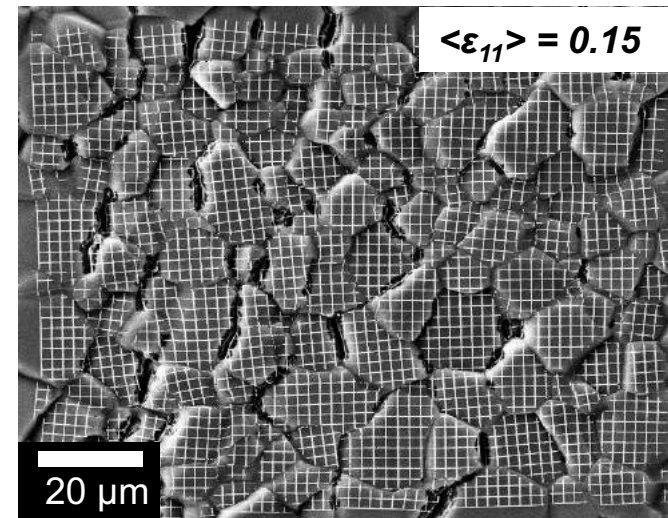
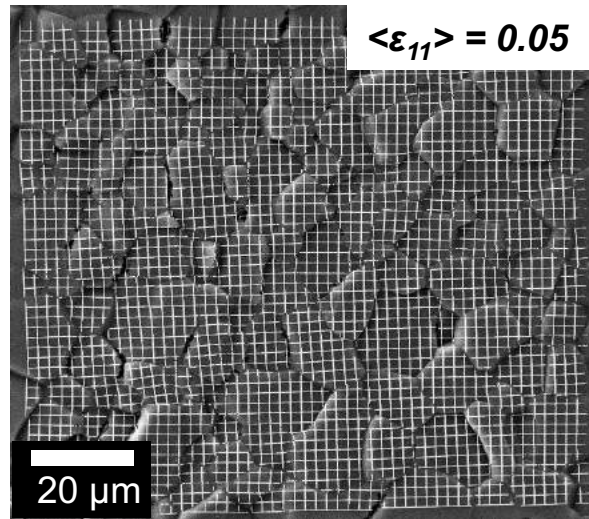
$\langle \epsilon_{11} \rangle = 0.05$

$\epsilon_{eq}$  normalized



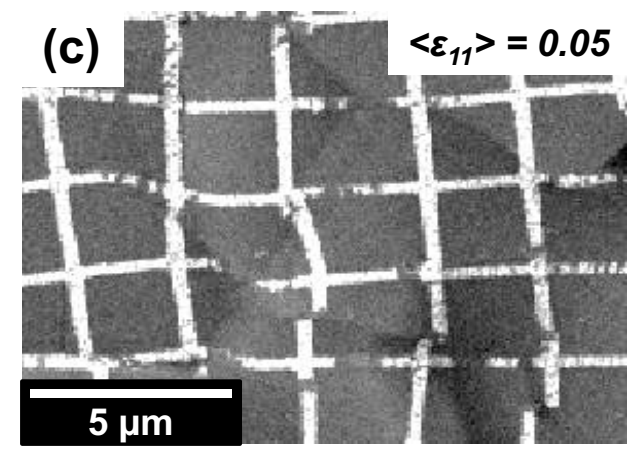
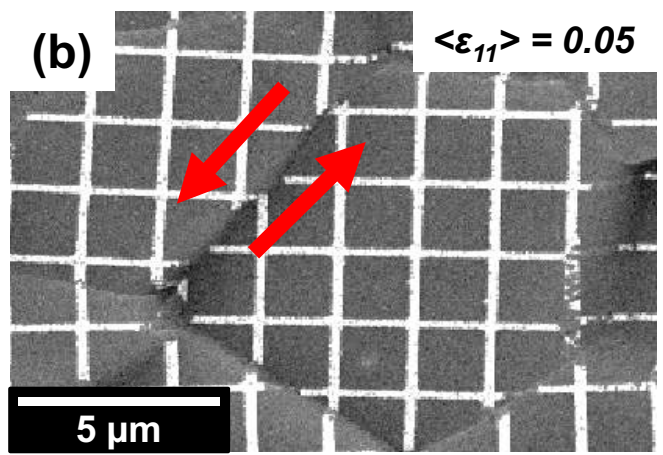
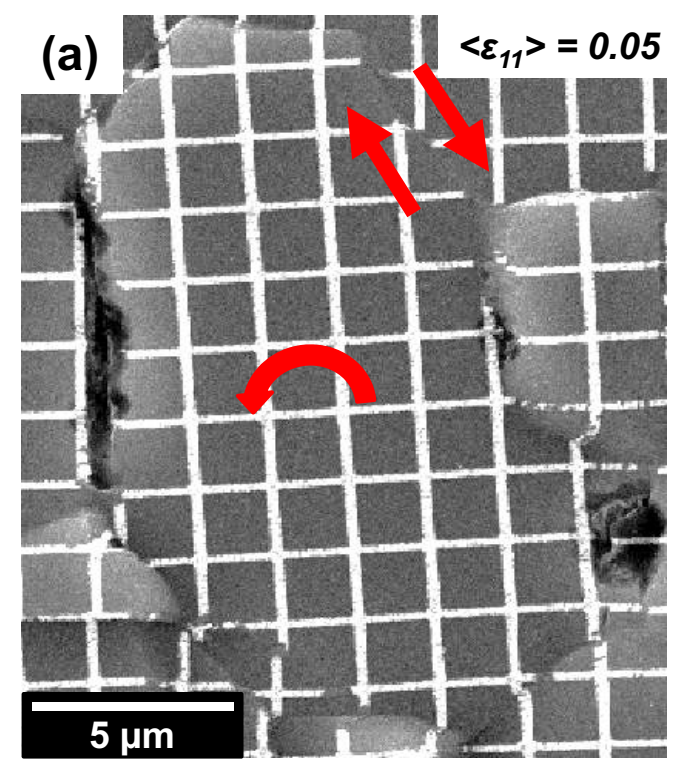
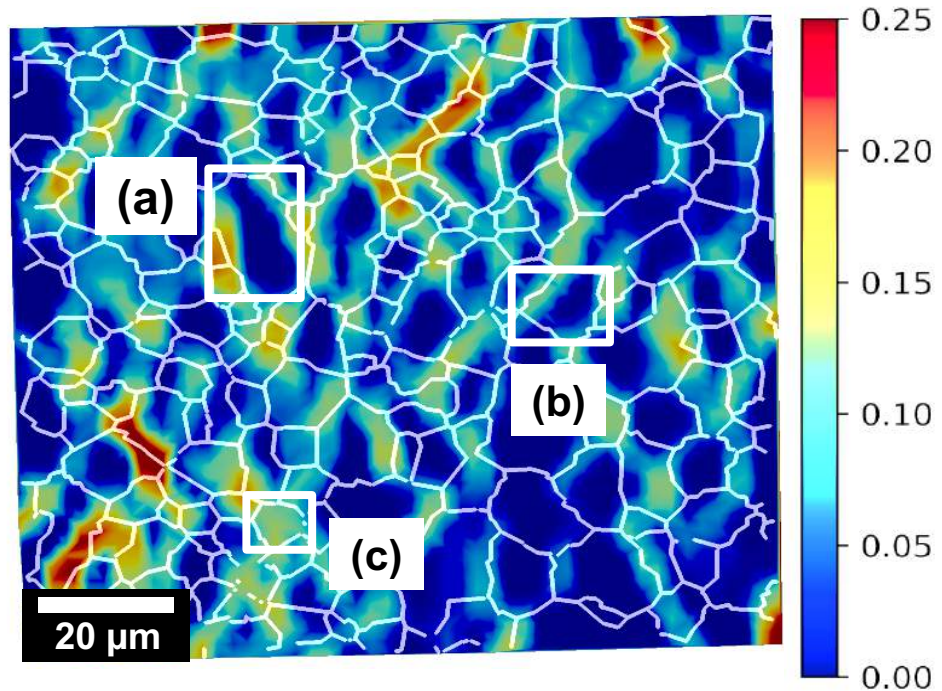
Strain map with superposition of GB deform

# Strain map



- ⇒ Presence of strain heterogeneities
- ⇒ Significant strain heterogeneities in some regions → Vicinity of grain boundaries
- ⇒ Low strain in the center of the grains

# Relation between strain map and deformation mechanisms



# Outlook

## Conclusion

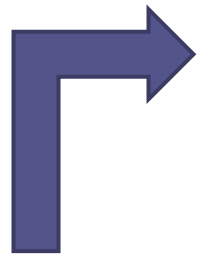


**We just showed the development work for an in situ tensile test at high temperature within SEM on Mg-alloys**



**Development of a « toolbox » for quantifying the different deformation mechanisms**

- **Different deformation mechanisms function to temperature**
- **Compare heterogeneities of deformation of our results at different temperatures**
- **Quantification of inter and intragranular component**



**Conference : Mg 2018 – Old Windsor (UK) in July 2018**

*The 11th International conference of Mg alloys and their applications (Mg 2018)*

***Paper in progress***

# Thanks for your attention Any questions ?

05/06/2018

**Dessolier Thibaut**

PhD student

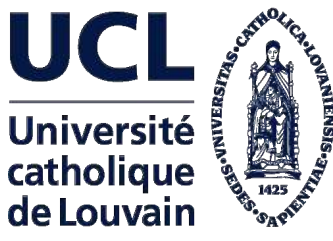
SIMaP Laboratory - GPM2 group

*Thibaut.dessolier@simap.grenoble-inp.fr*

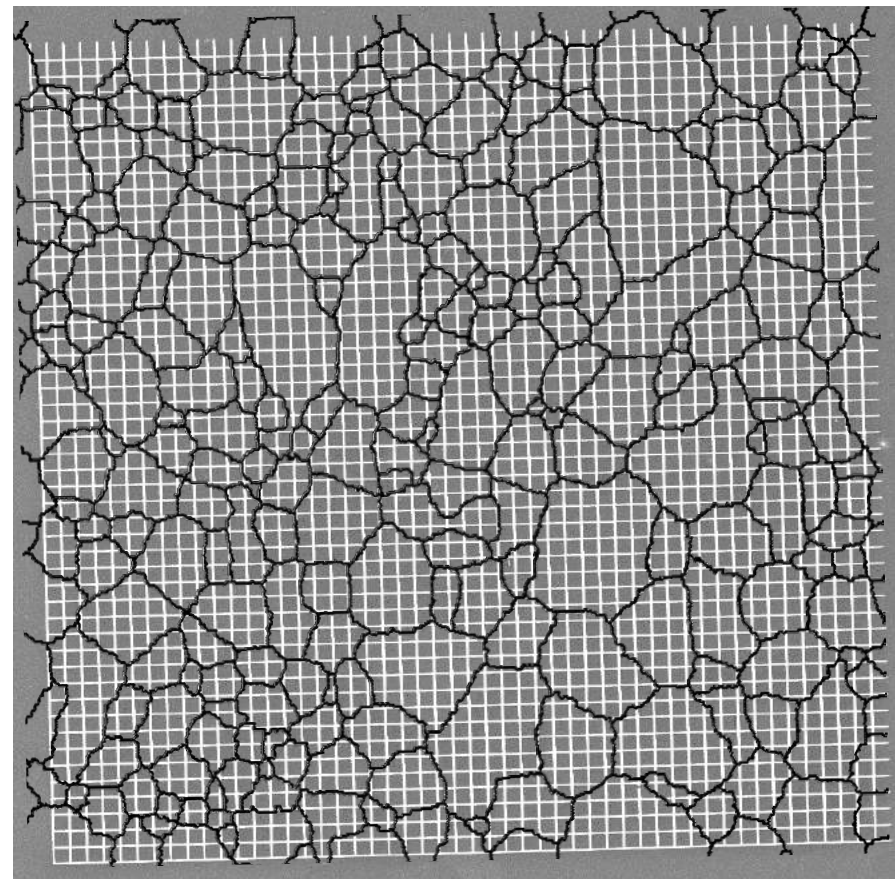
Thesis supervisor : Blandin Jean Jacques

Advisors : Martin Guilhem and Lhuissier Pierre

Collaboration : Delannay Laurent

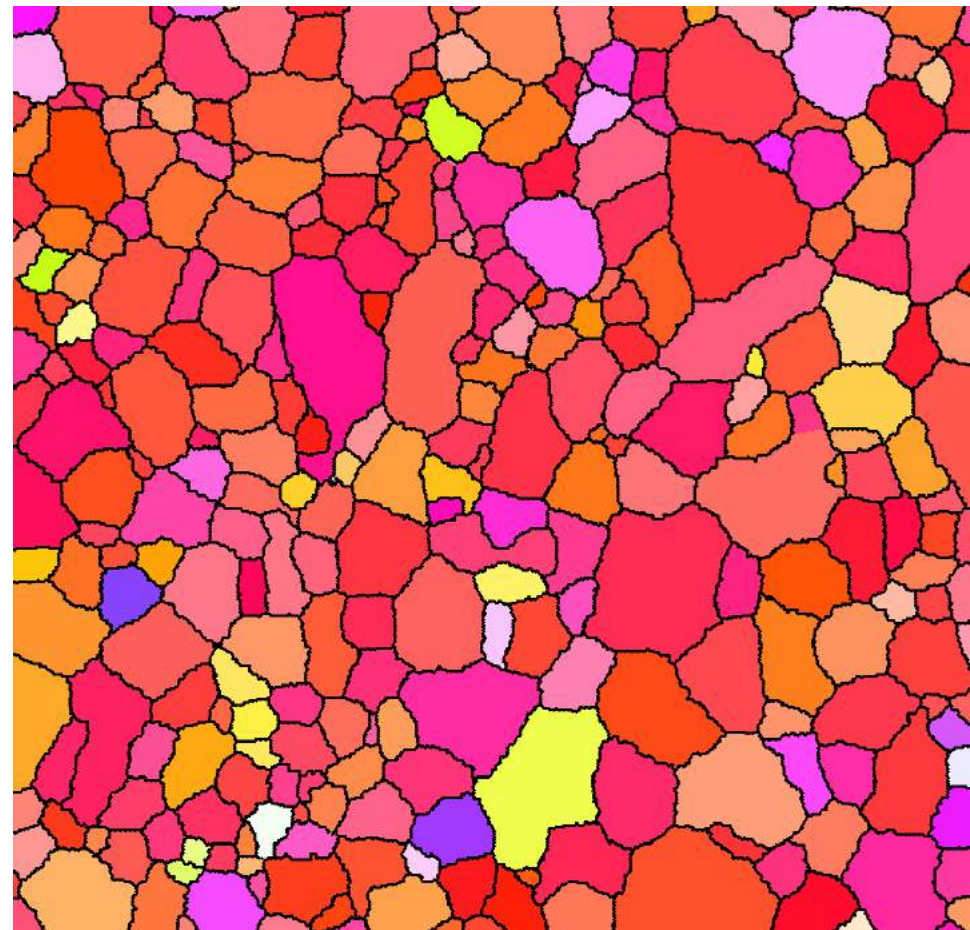
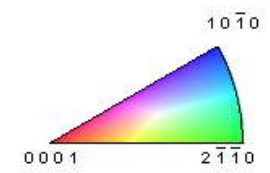


# Superposition



20 μm

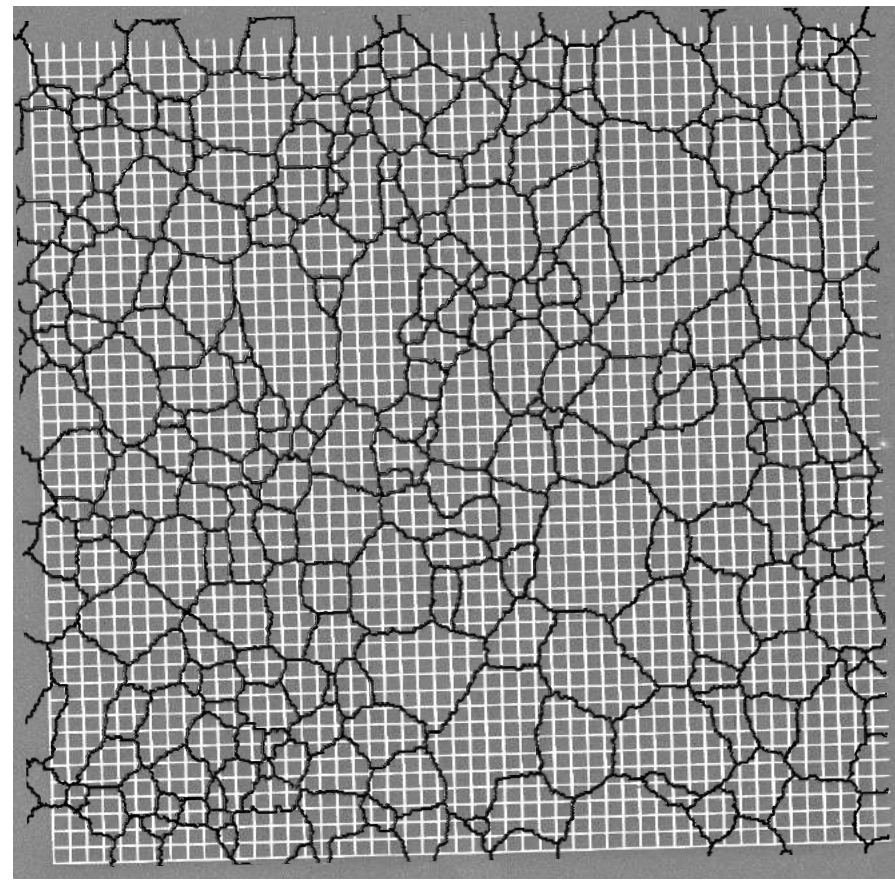
$$\langle \epsilon_{11} \rangle = 0$$



20 μm

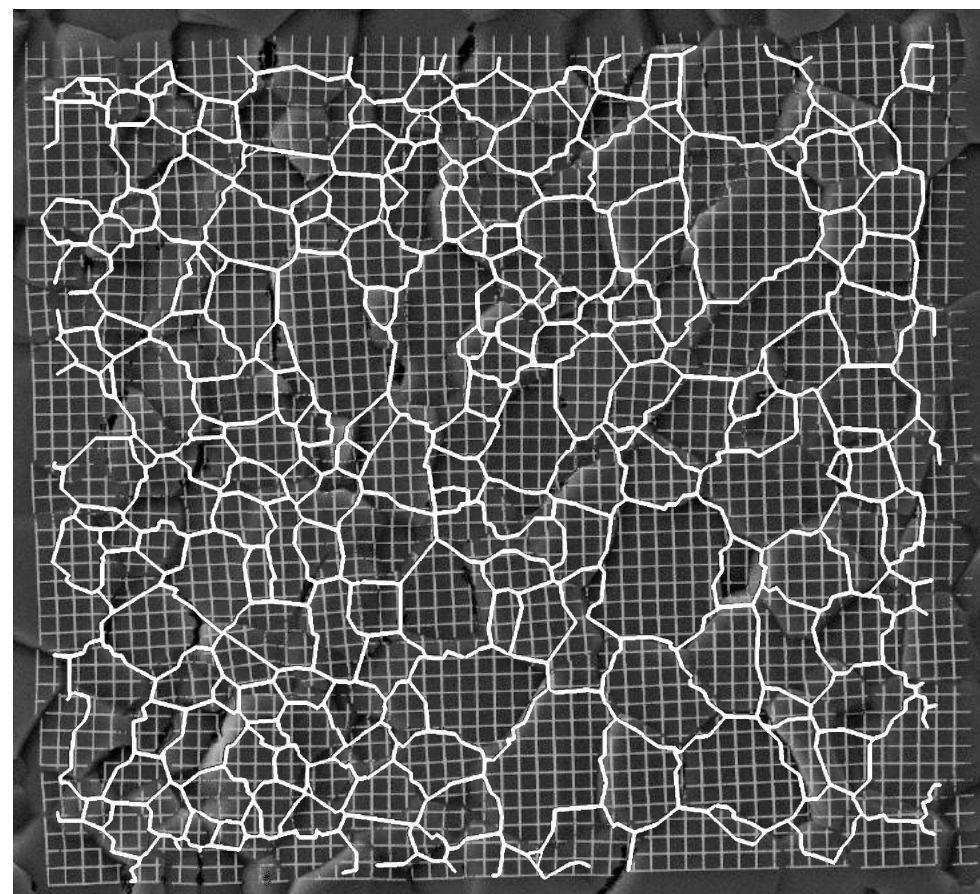
IPF EBSD-map before lithography deposition

# Superposition



20 μm

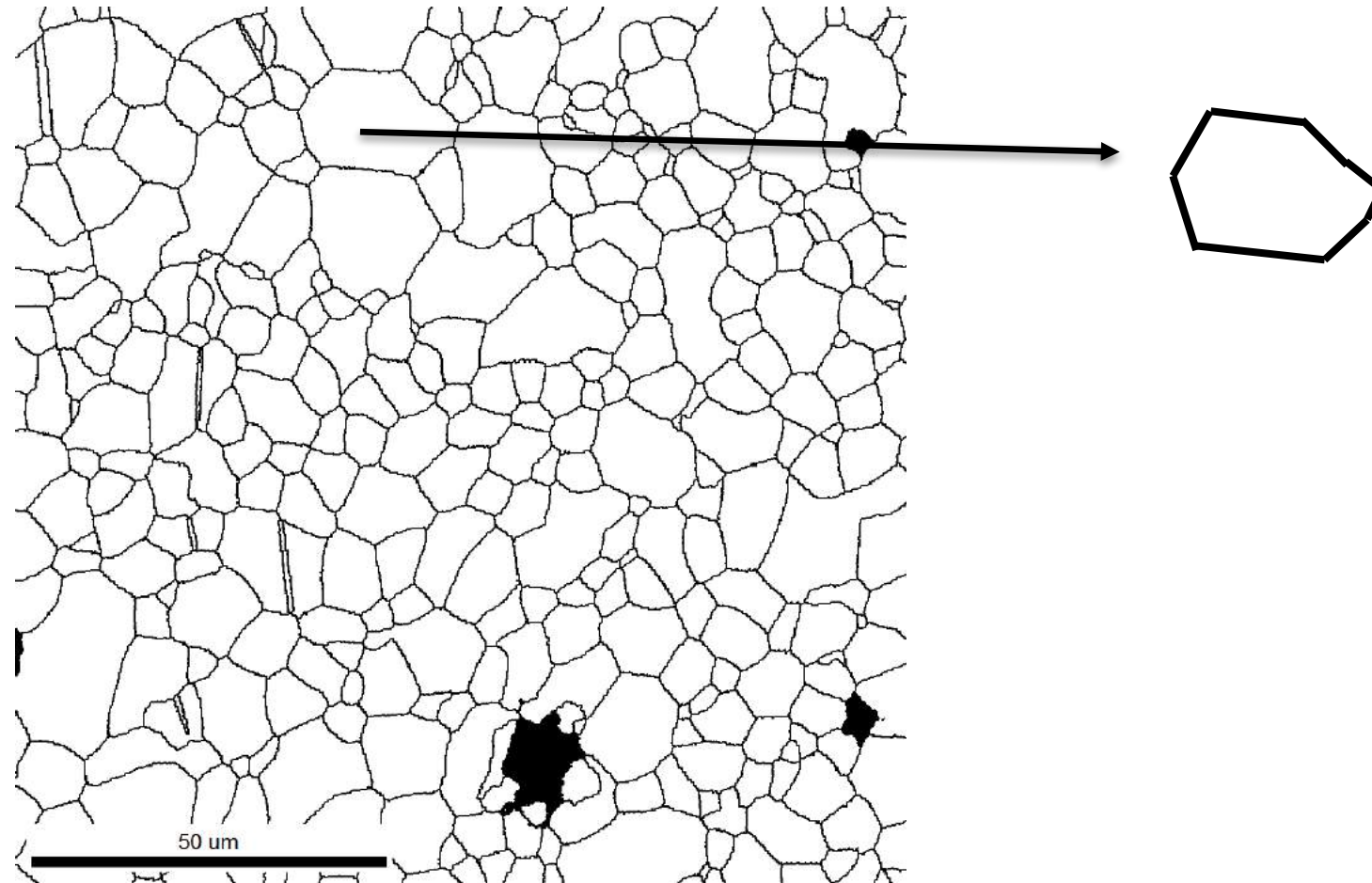
$$\langle \epsilon_{11} \rangle = 0$$



20 μm

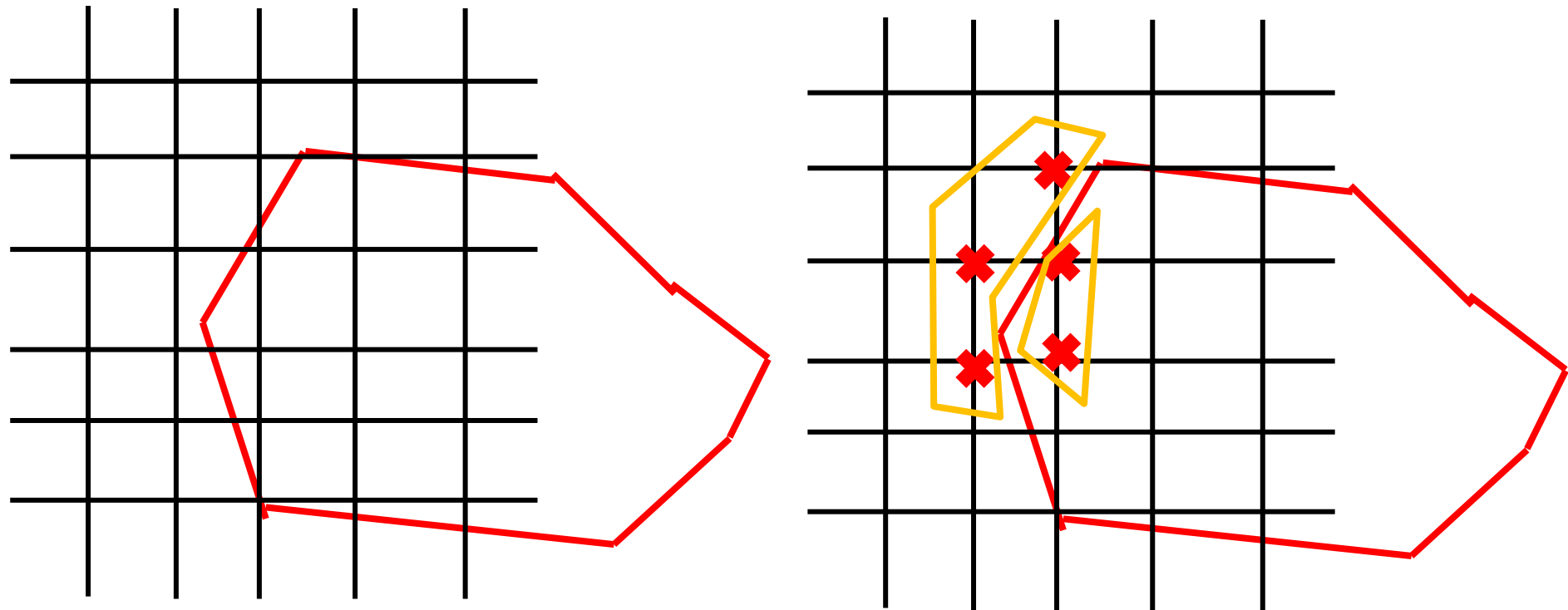
$$\langle \epsilon_{11} \rangle = 0.05$$

## Approximation



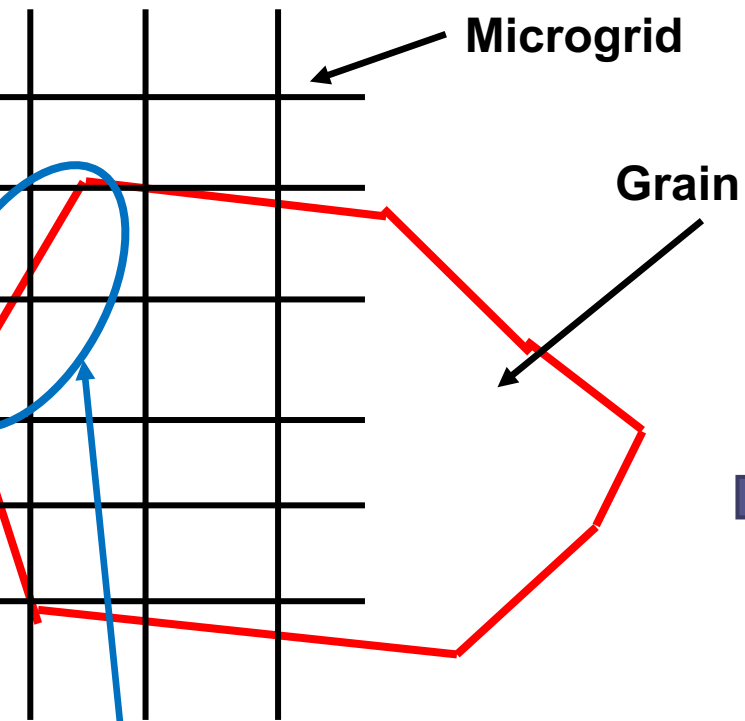
Line approximation

## Approximation

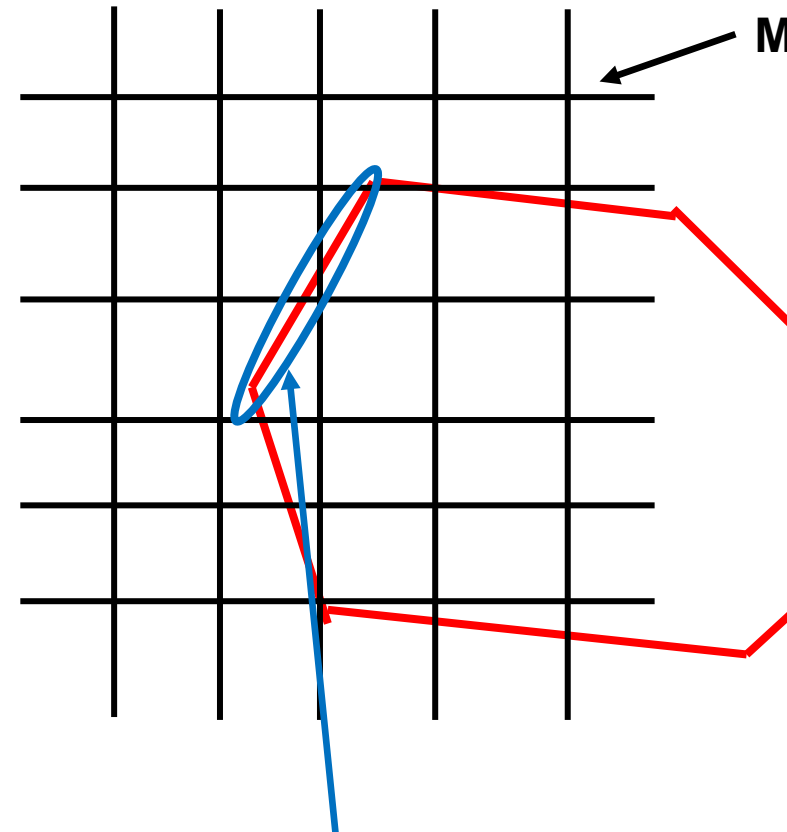


Define points

## deformations mechanisms



the area we calculate Slide and Open  
 Not just the GB  
 We have a part of the grain inside



Refine the area

