



UNIVERSITÉ
DE LORRAINE



Time resolved and *in situ* characterization of recrystallization during intercritical annealing of cold-rolled steels

07.06.2018

ALEMI 2018 - METZ

M.MORENO
S.ALLAIN
J.TEIXEIRA
F.BONNET

Microstructural design of the studied Dual-Phase steel: Microstructure / Property Relationships

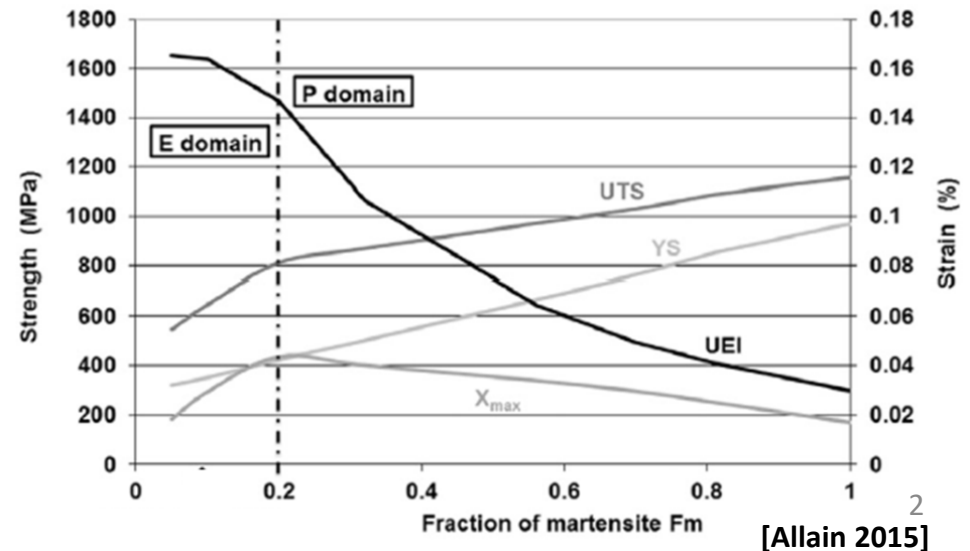
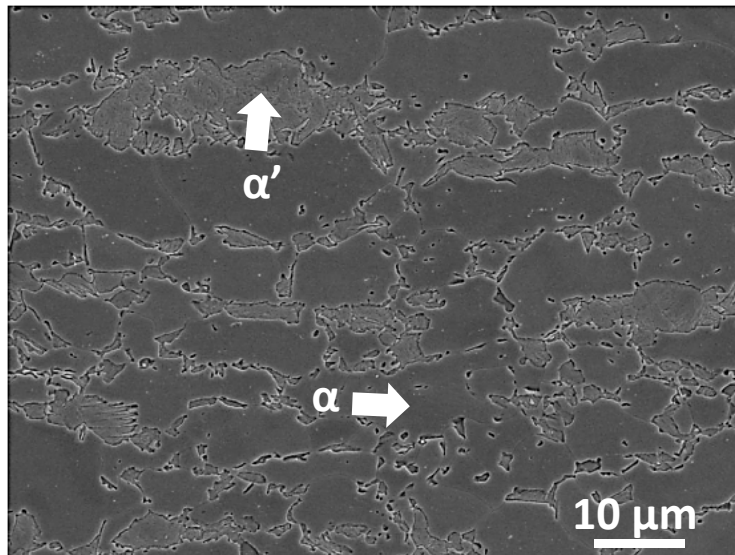
- Dual-Phase (DP) are extensively used in the automotive industry



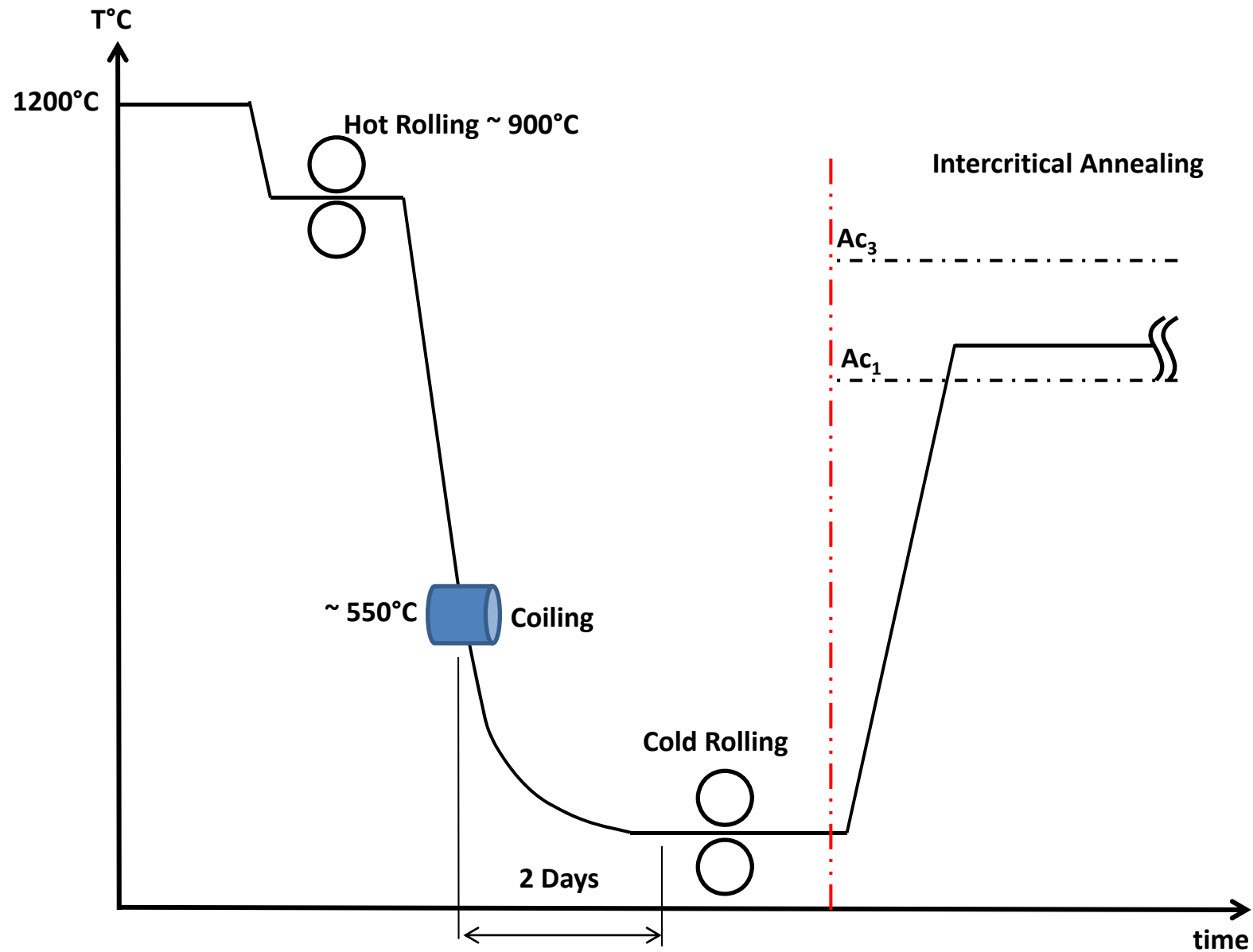
Cold Drawing / Stamping

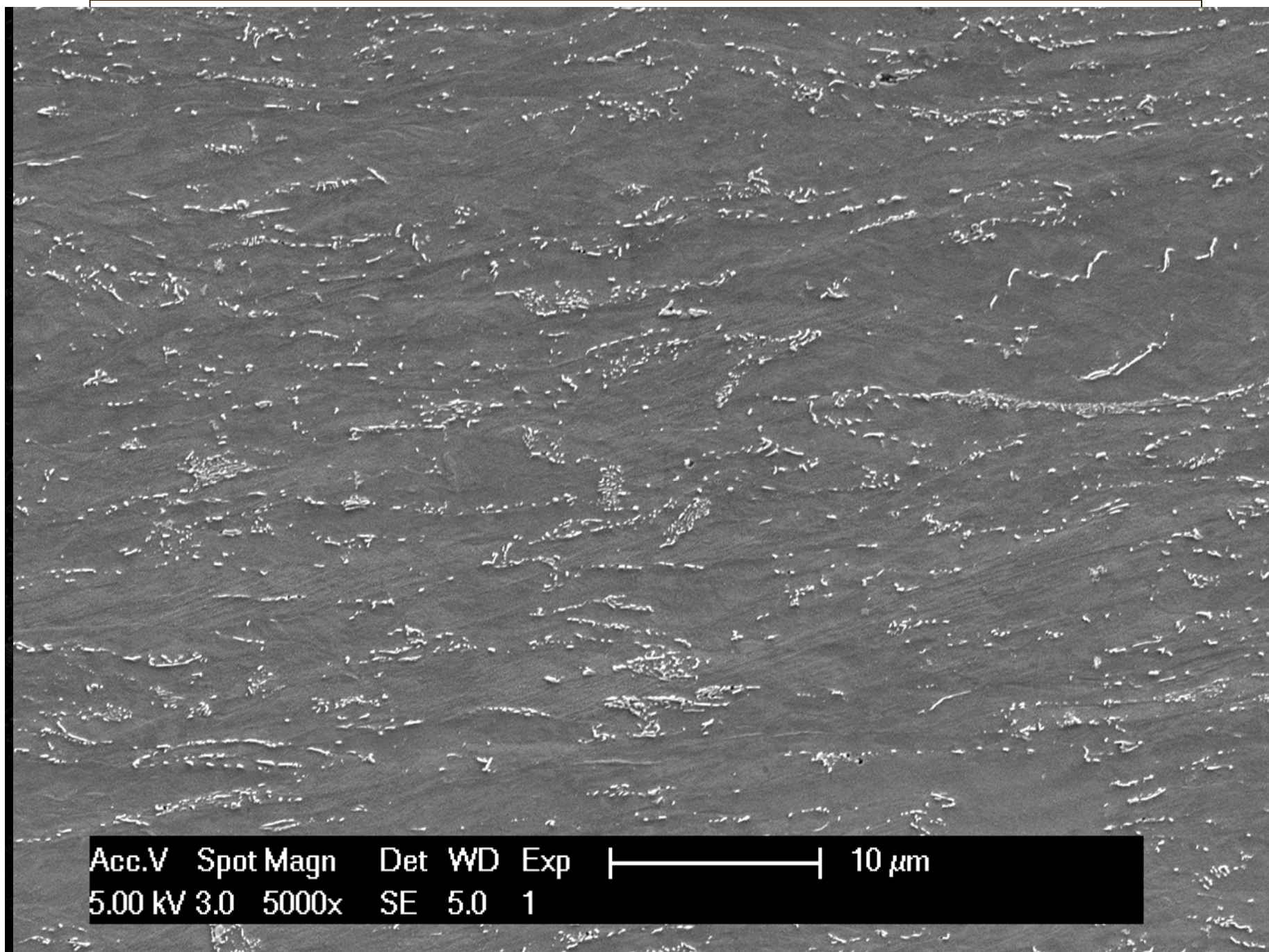


- DP = heterogenous microstructures made of α and α' phases (typically 10 - 25 %vol in DP600-DP800)
- Good balance between strength and ductility
- Mechanical properties strongly related to the fraction of α'
- Important to control martensite fraction of DP steels



Microstructural design of the studied Dual-Phase steel: Production

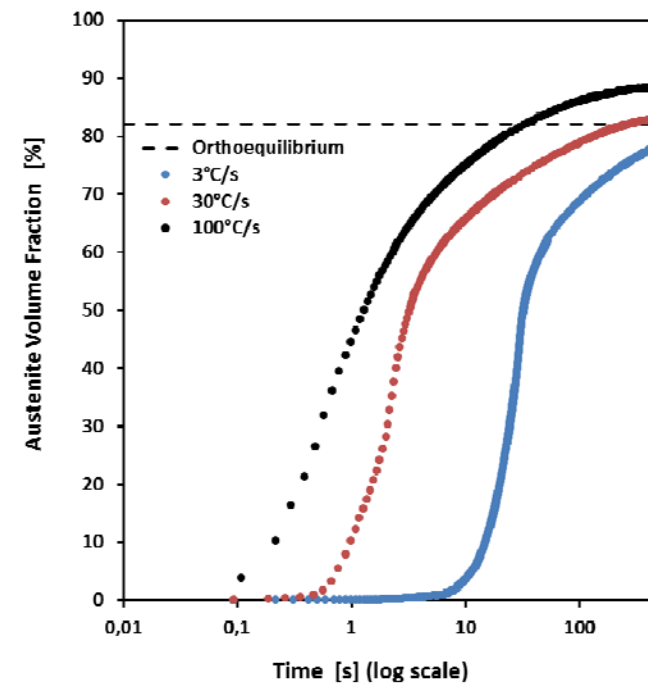
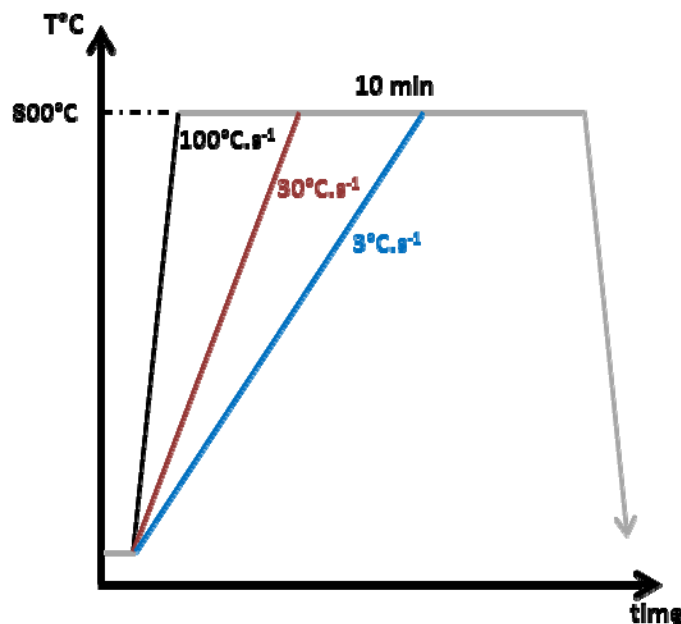




Acc.V	Spot	Magn	Det	WD	Exp	Scale Bar
5.00 kV	3.0	5000x	SE	5.0	1	10 μm

Microstructural design of the studied Dual-Phase steel: Intercritical Annealing

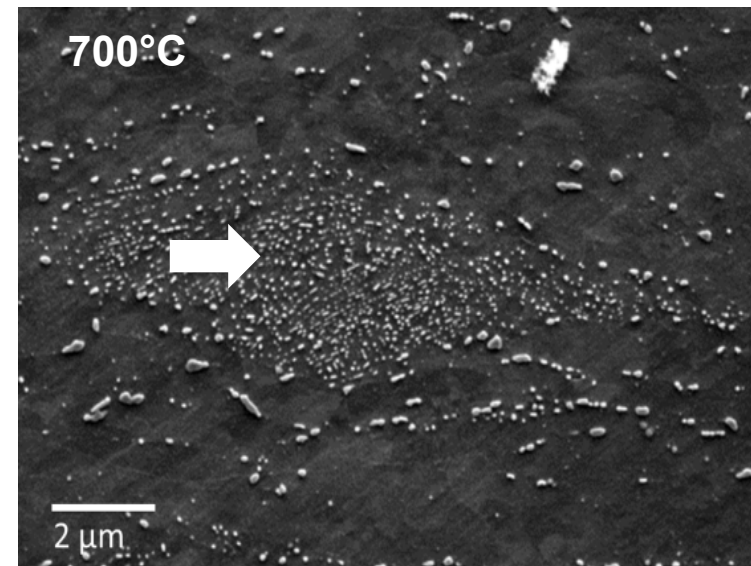
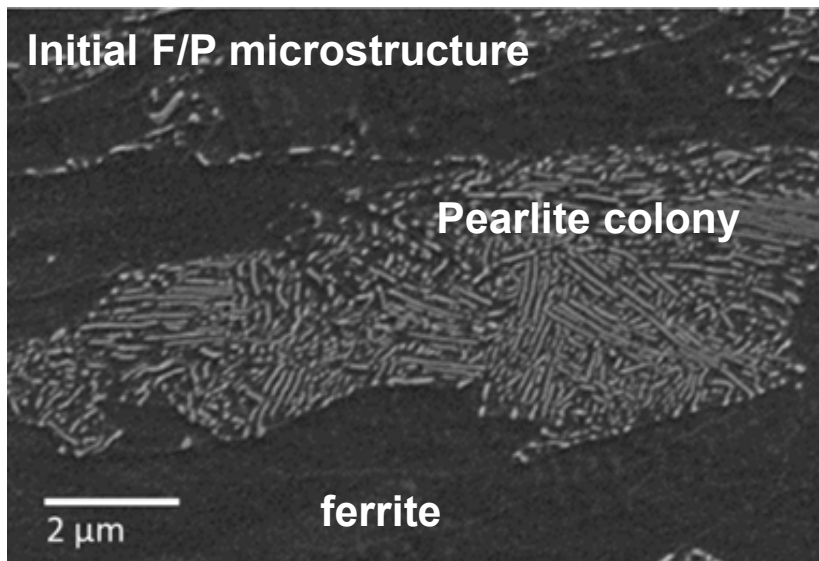
- Martensite fraction depends on intercritical annealing conditions
- Austenitization affected by different mechanisms occurring during heating of cold rolled ferrite/pearlite microstructure
 - Fragmentation / Spheroidization / Ripening of cementite lamellae
 - Recovery / Recrystallisation of ferritic matrix



- Huge impact of the morphology/composition on the austenitization kinetics

Microstructural design of the studied Dual-Phase steel: Intercritical Annealing

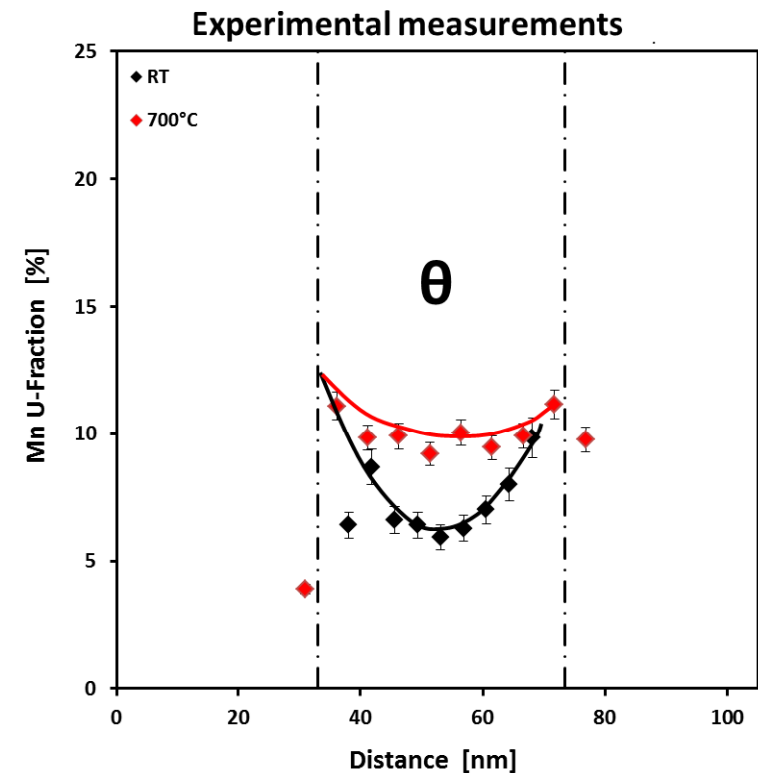
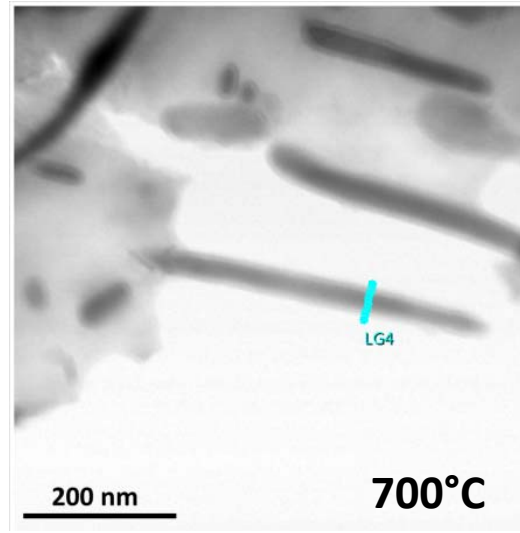
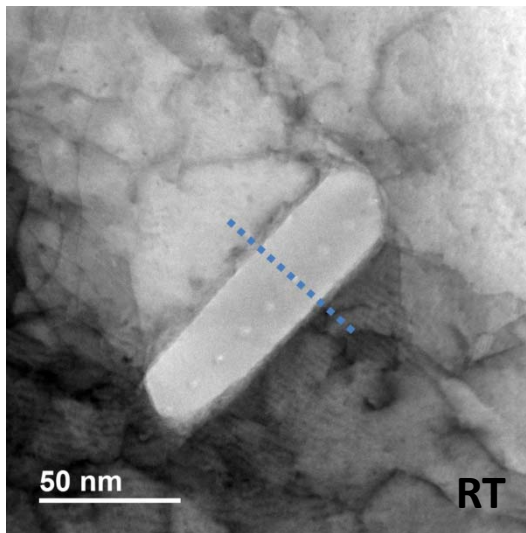
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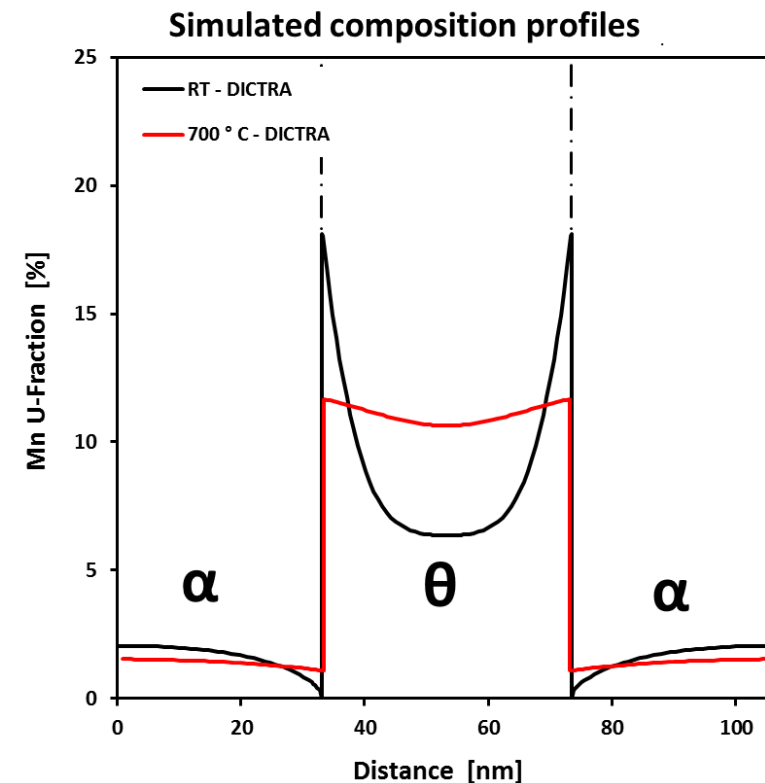
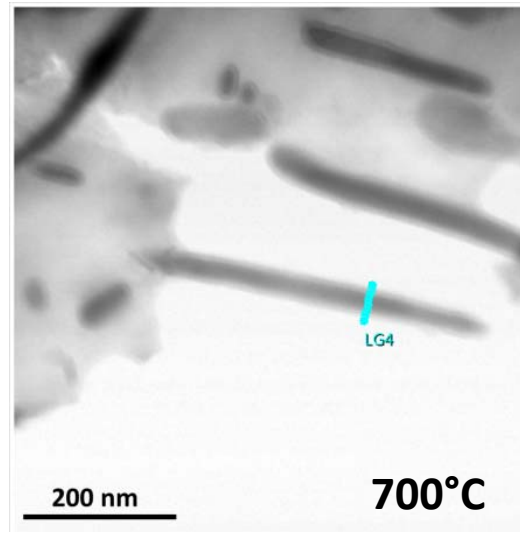
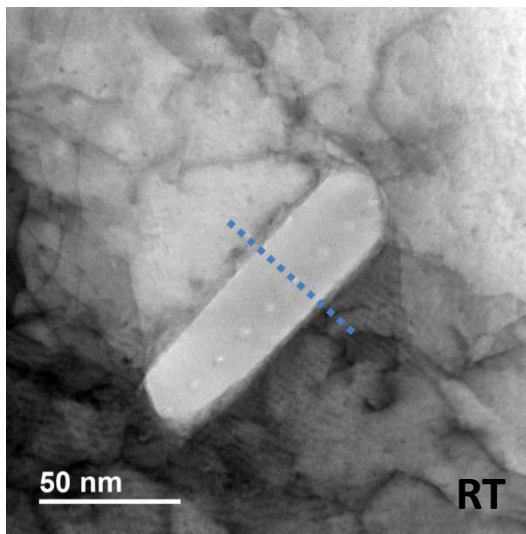
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Microstructural design of the studied Dual-Phase steel: Intercritical Annealing

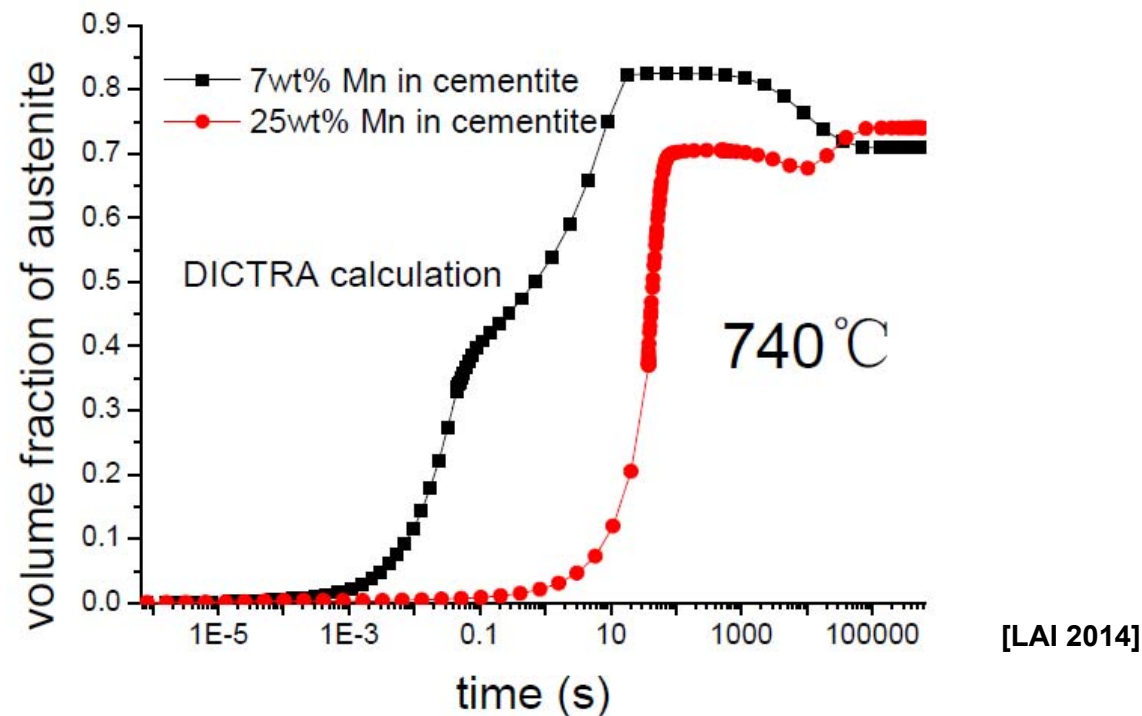
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- DICTRA & Reassessment of Mn diffusion coefficient in cementite (to be published soon)

Microstructural design of the studied Dual-Phase steel: Intercritical Annealing

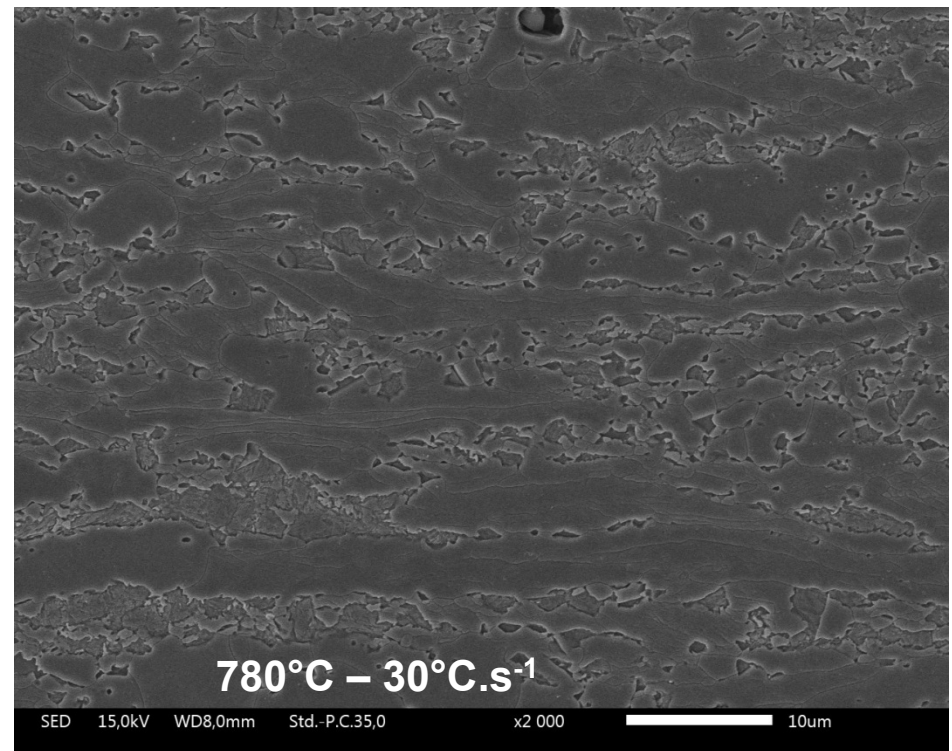
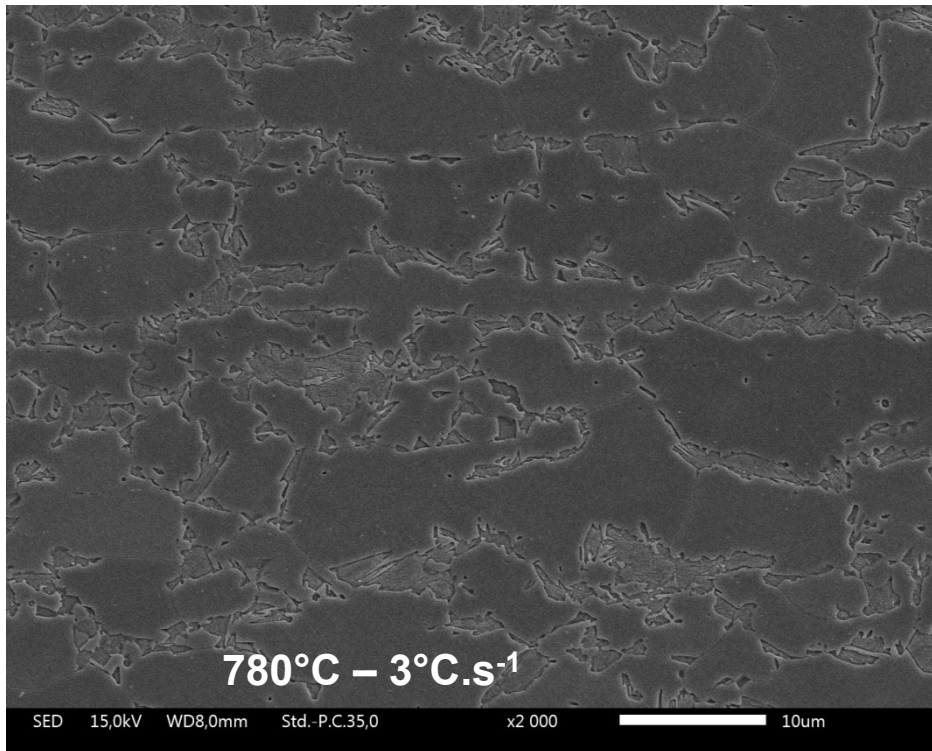
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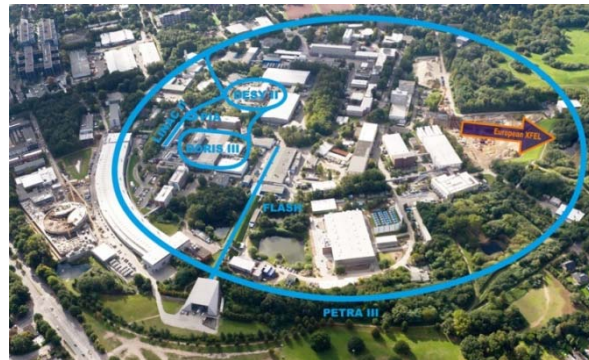
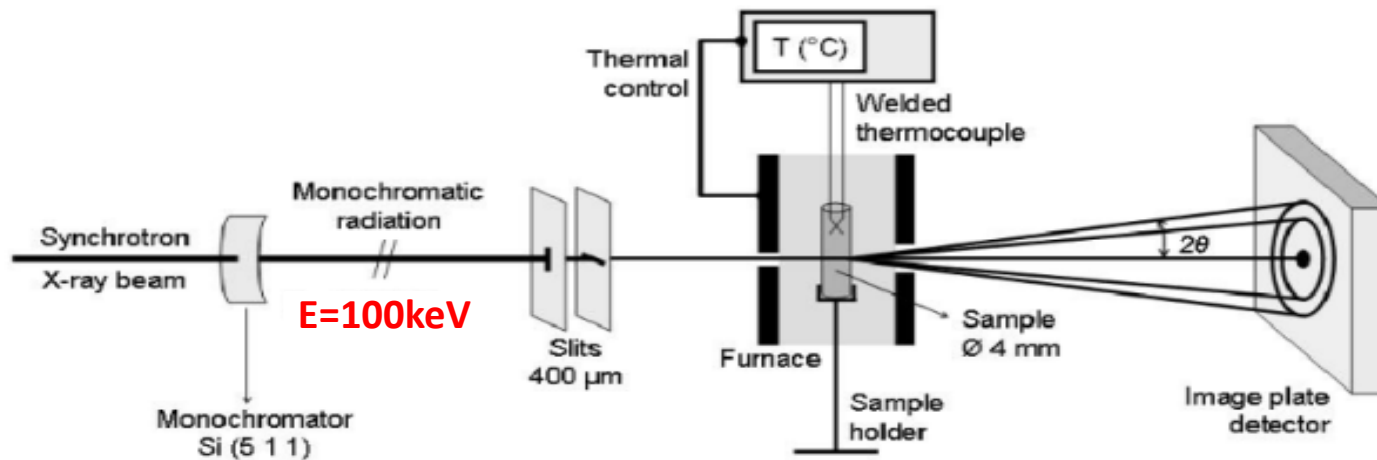
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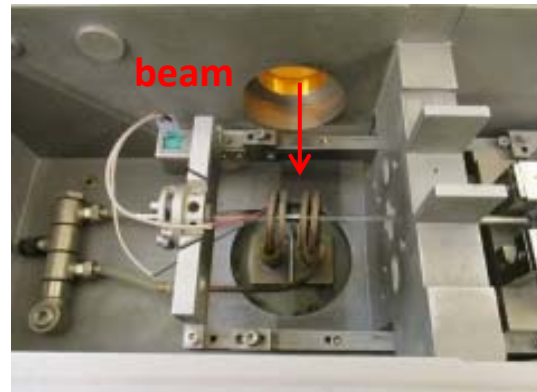


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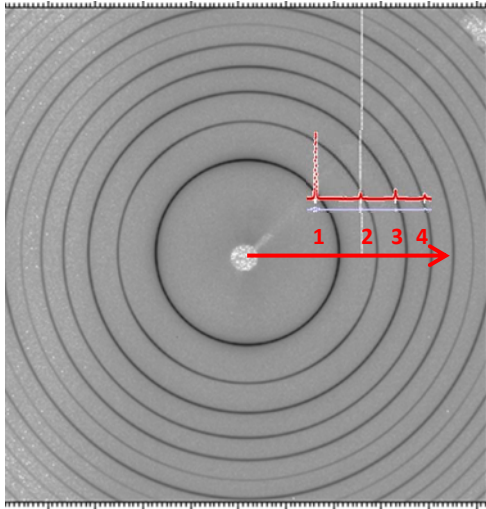
HEXRD – Experimental setup



- Monochromatic beam 100 KeV
- Diffraction in transmission
- Powder diffraction condition
- High acquisition frequency: 10 Hz
- Sample environment: Bähr dilatometer



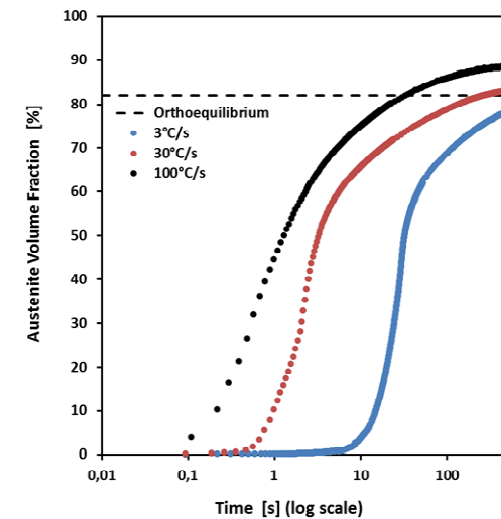
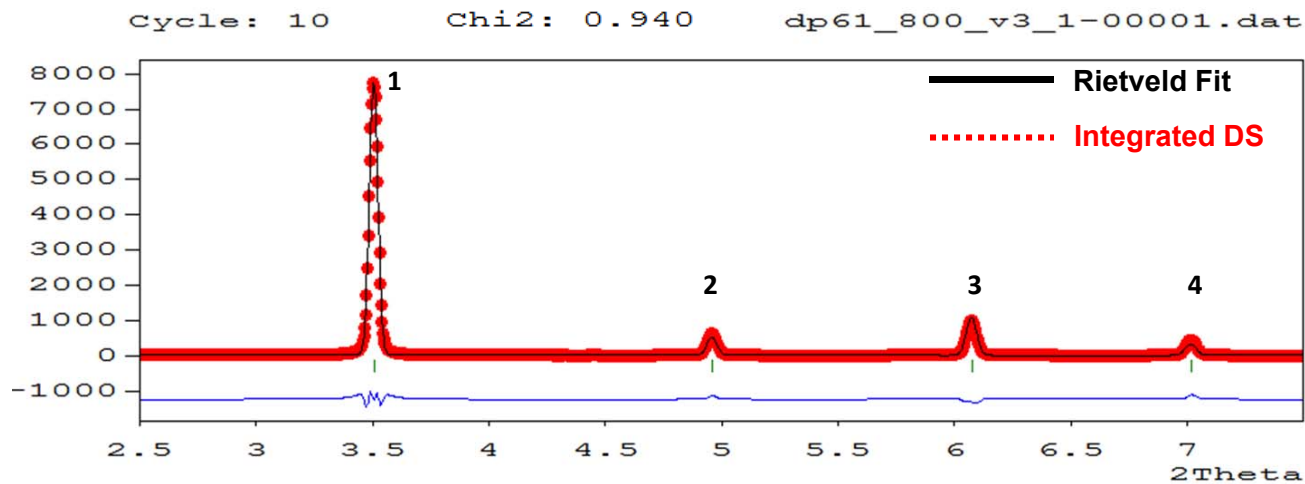
HEXRD Experiments



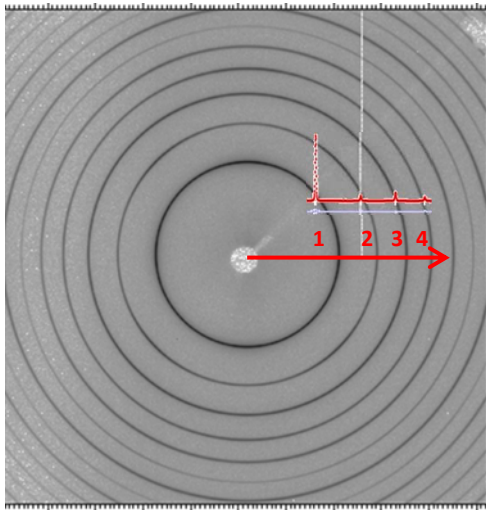
Numerous informations provided by HEXRD experiments: **Rietveld Analysis**

1. Phase transformation:

- Phase mass fraction
- Evolution of the lattice parameters



HEXRD Experiments



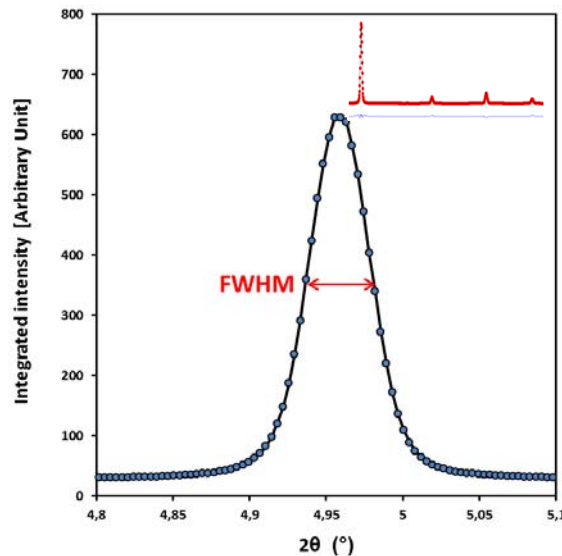
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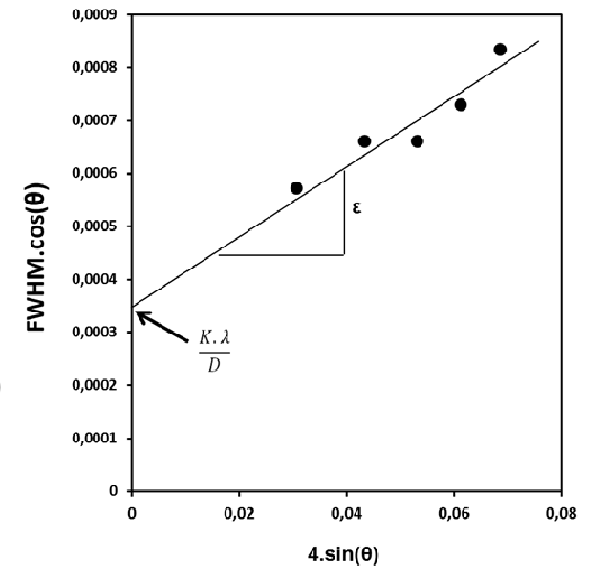
- Full Width Half Maximum
- Crystallite size D and the strain parameter ε of the material with Williamson-Hall method
- Estimation of the dislocation density with the formula of Smallman



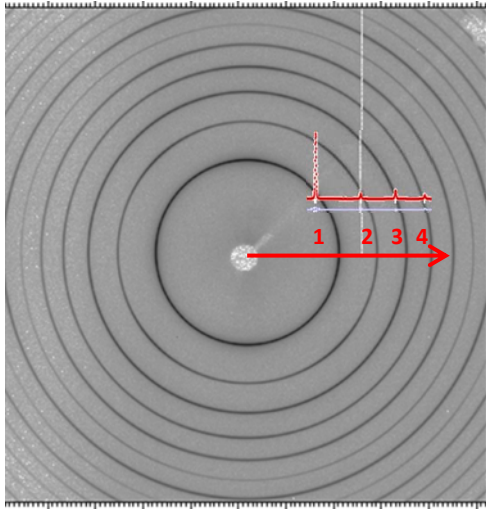
Equation of Williamson-Hall [WH 53]:

$$FWHM(\theta) \cdot \cos\theta = \frac{K \cdot \lambda}{D} + 4 \cdot \sin\theta \cdot \varepsilon$$

- θ = peak angular position (rad)
- λ = Synchrotron beam wavelength (m)
- D = Crystallite size (m)
- ε = strain parameter
- K = parameter (~ 0.9)



HEXRD Experiments



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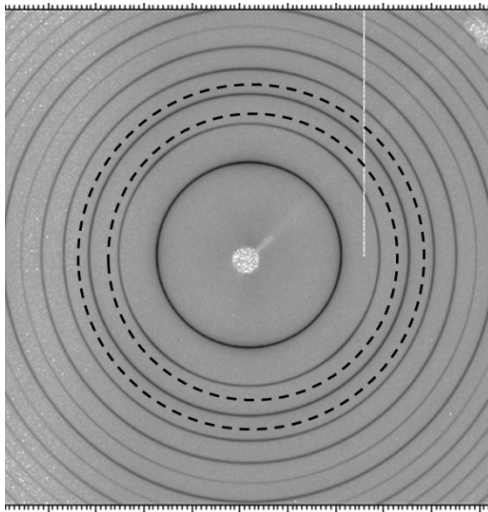
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Equation of Smallman [SM 57]:

$$\rho = \frac{3\sqrt{2\pi}\varepsilon}{bD}$$

- ρ = dislocation density ($\text{m} \cdot \text{m}^{-3}$)
- b = Burger's vector (m)
- D = crystallite size (m)
- ε = strain parameter

HEXRD Experiments



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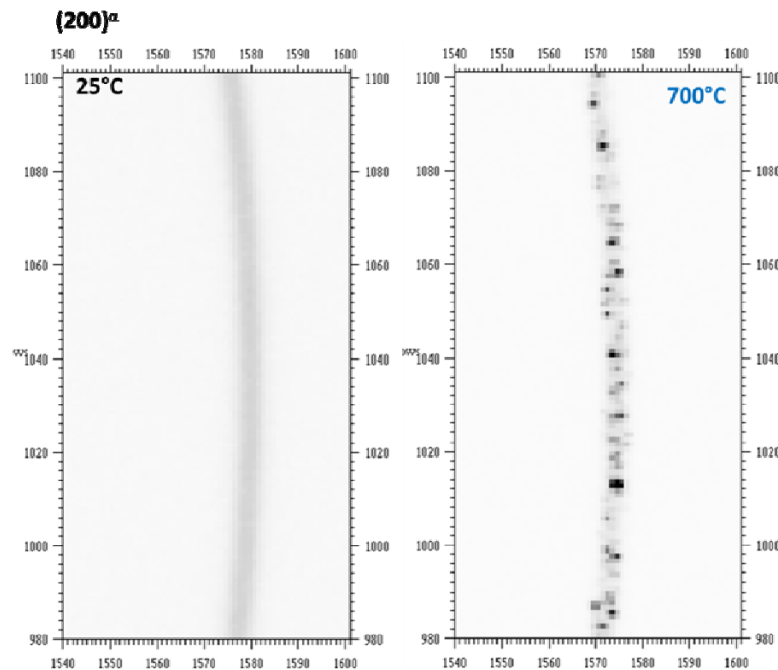
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3. Recrystallization

- Individual diffraction spots

$(200)^\alpha$ DS ring is not overlapped with:

- cementite
- austenite



HEXRD Experiments

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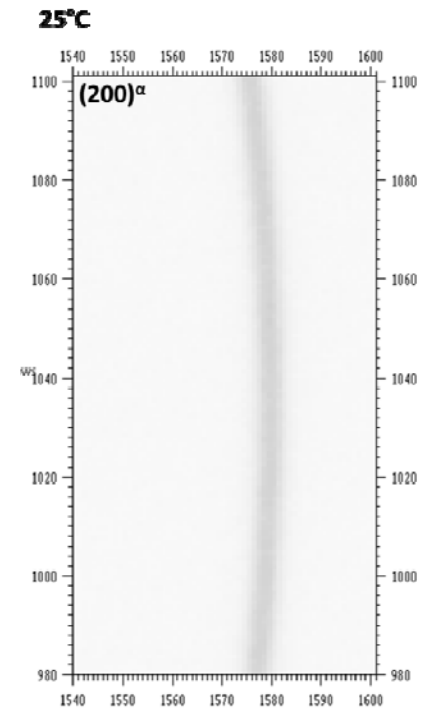
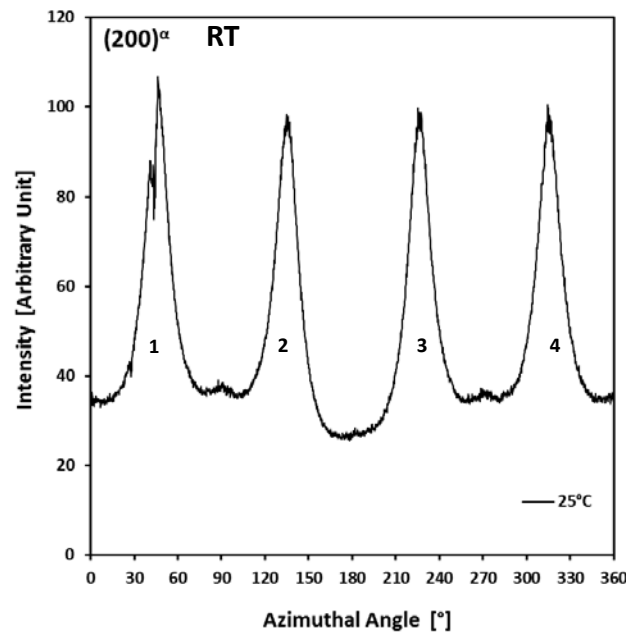
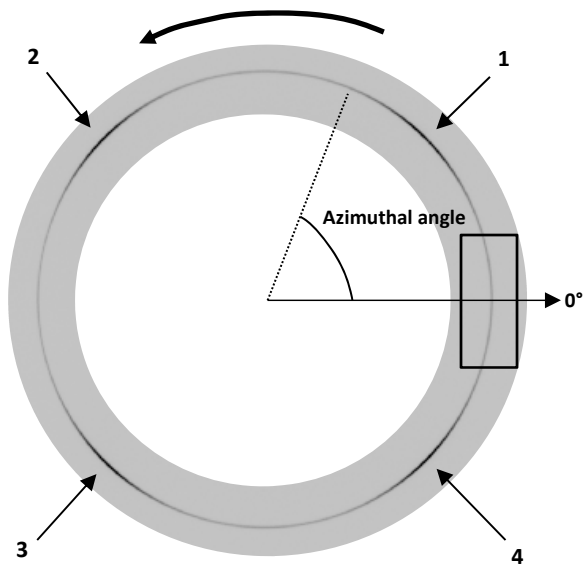
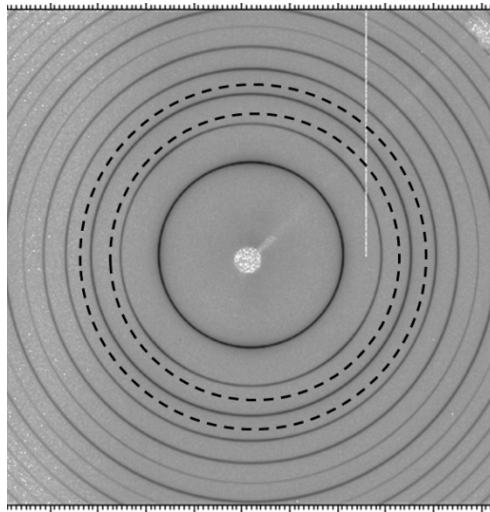
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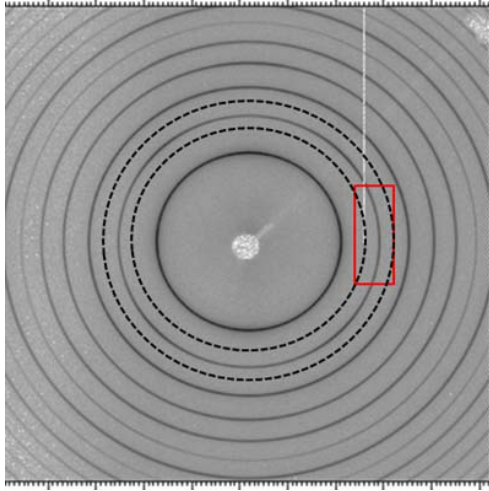
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HEXRD Experiments

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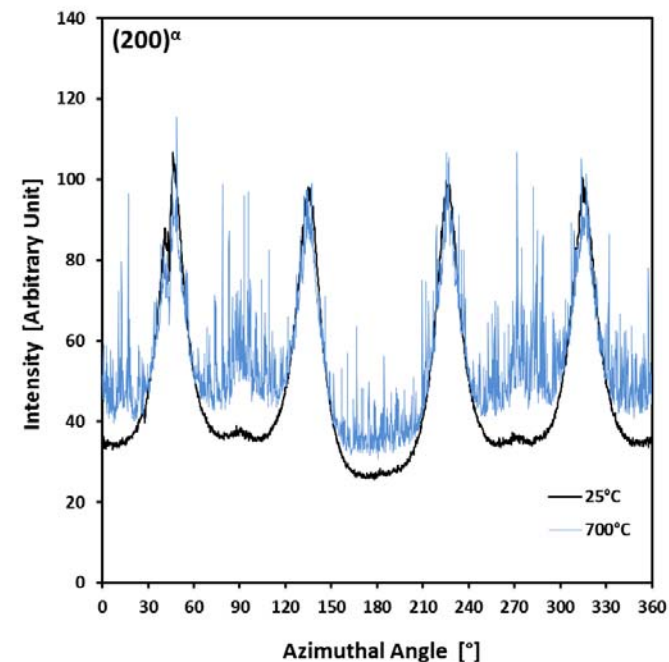
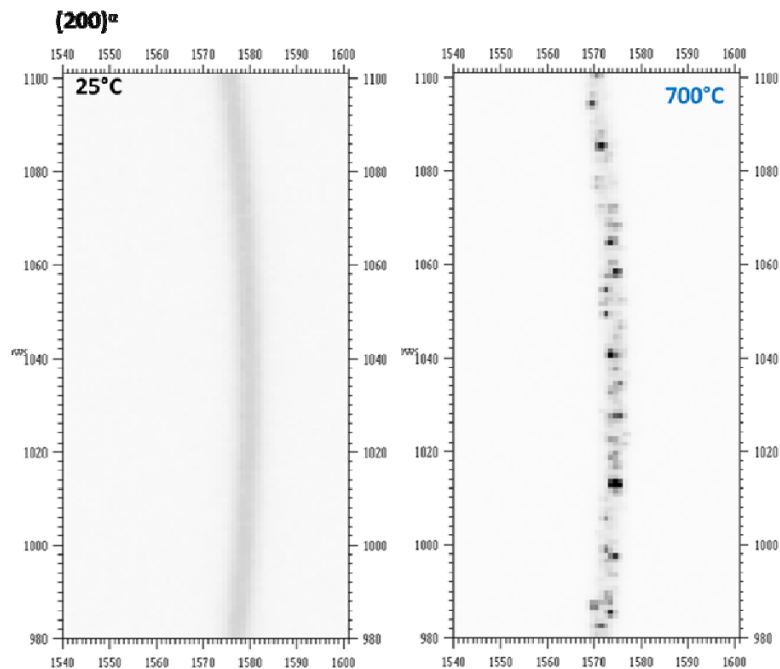
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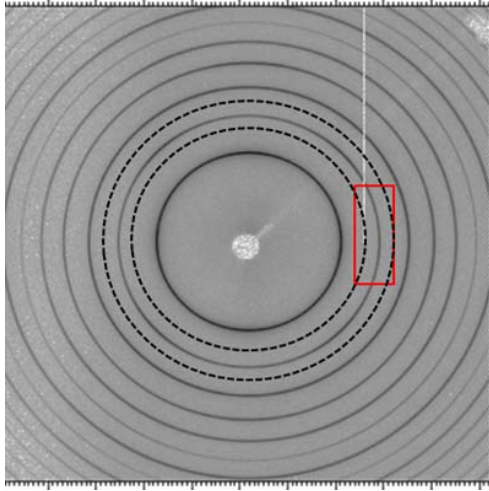
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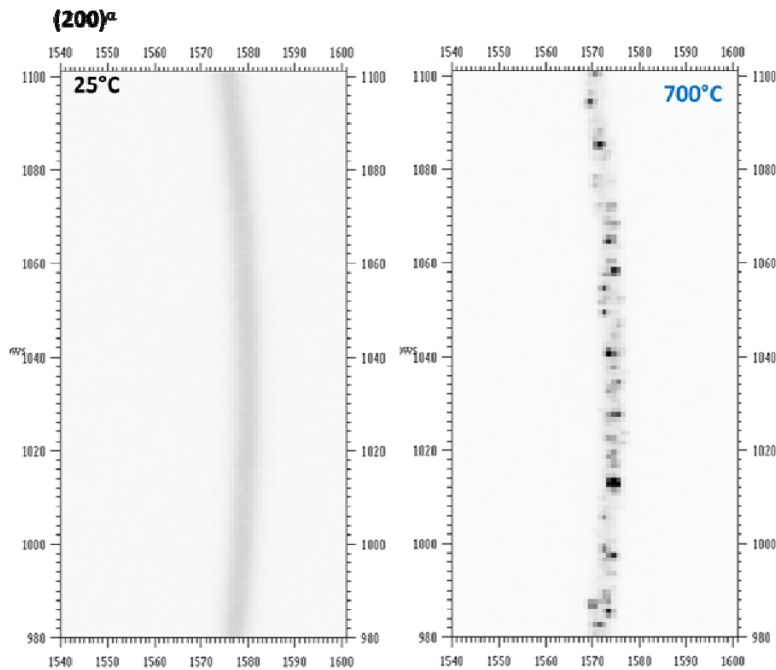
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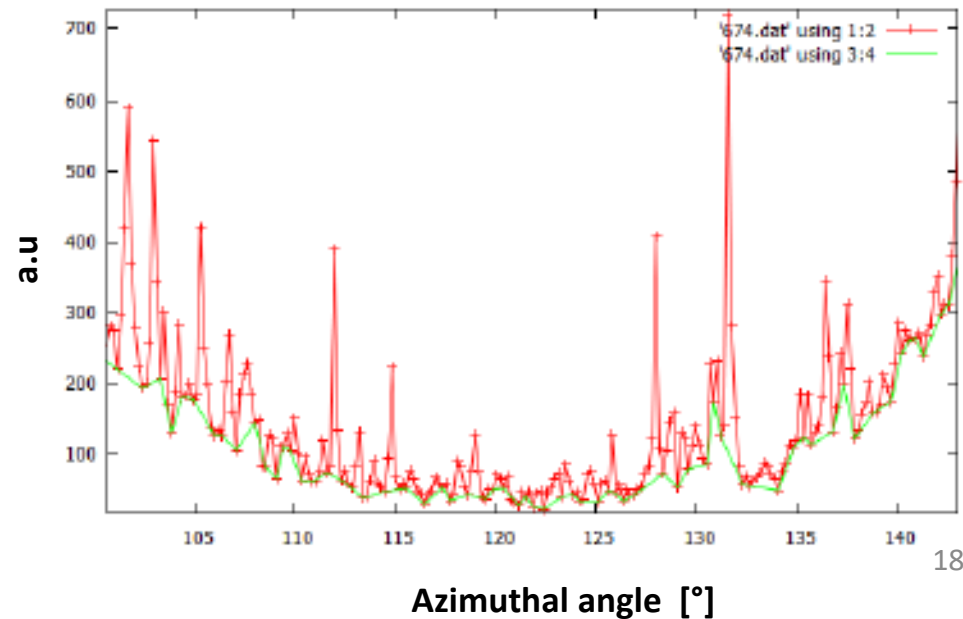
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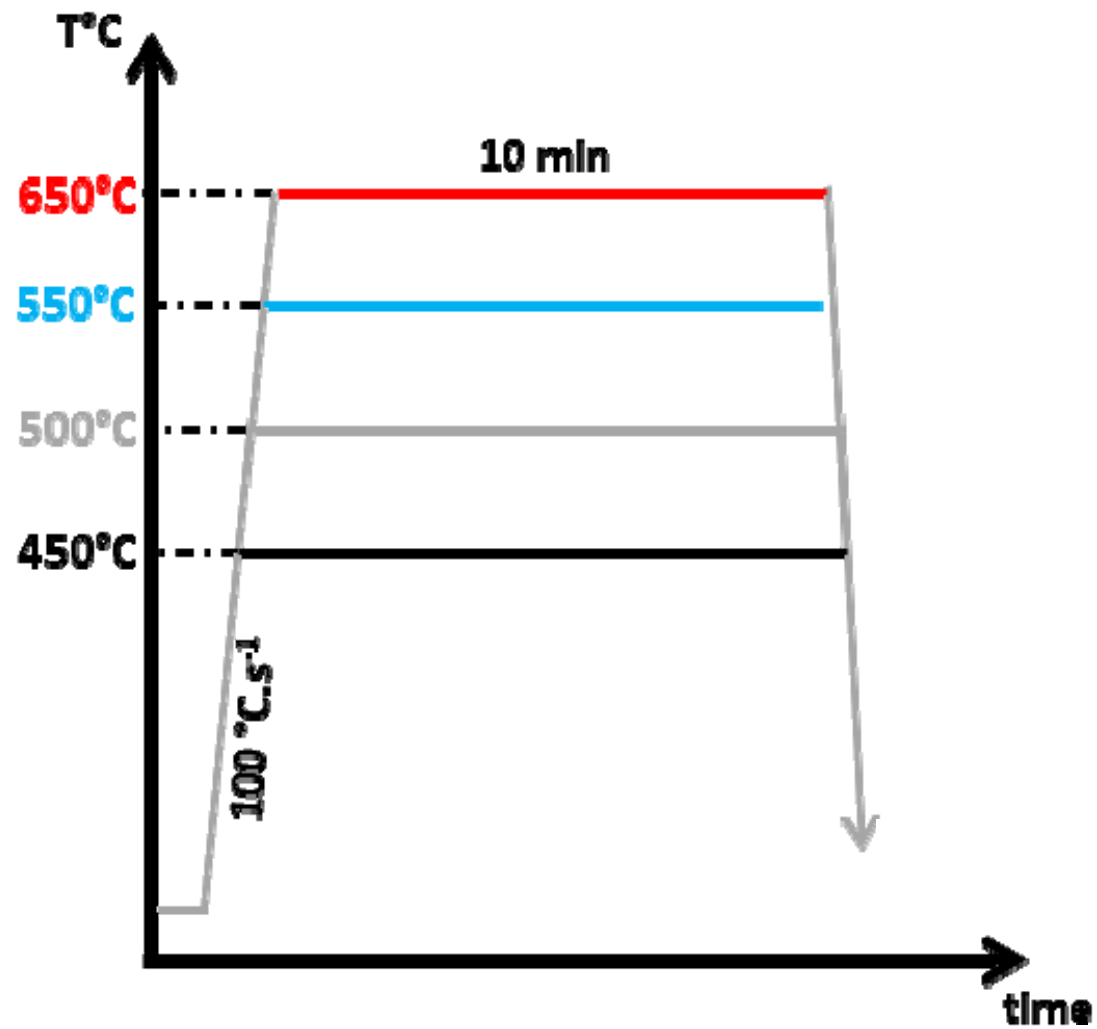


Development of an algorithm for spot counting

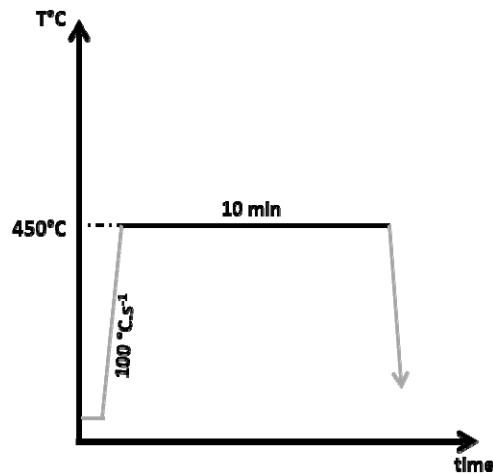


Application on model Fully Ferritic steel

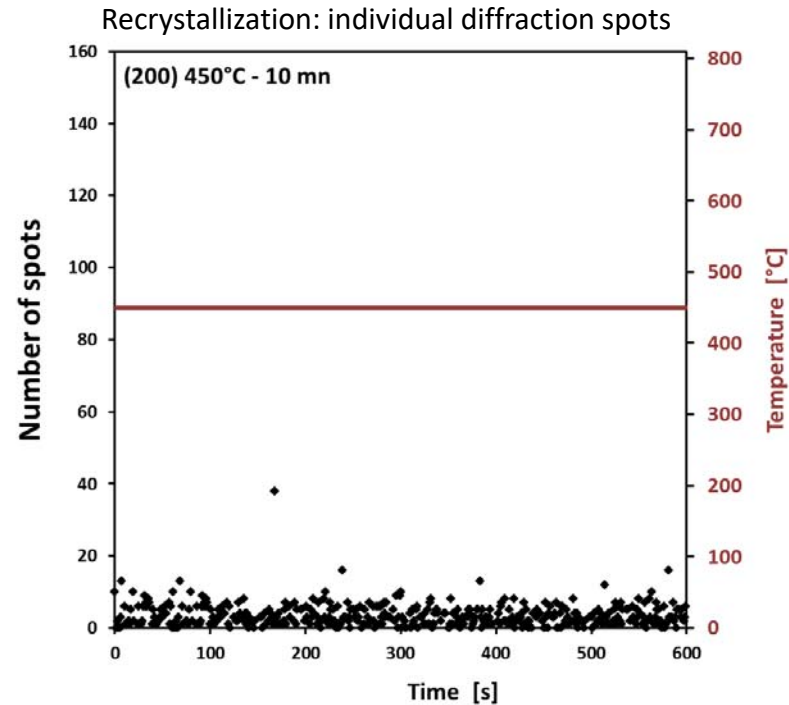
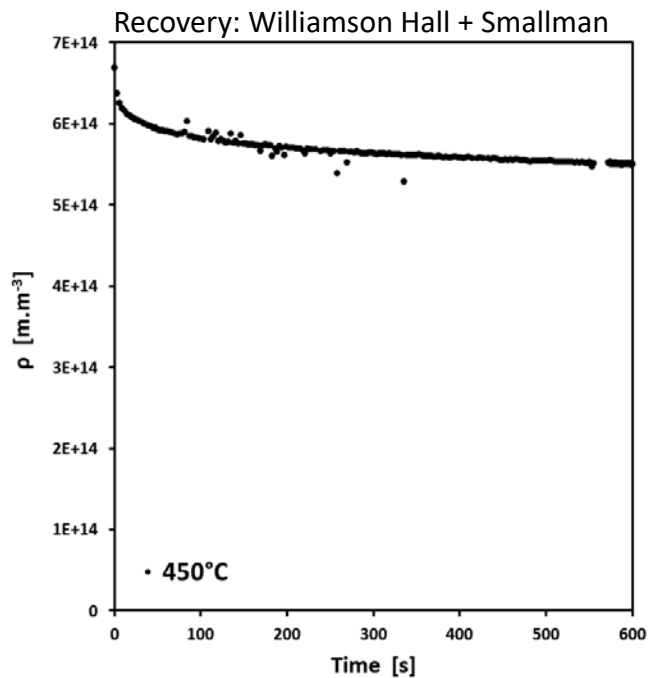
ULC Steel: no concomitant phase transformation, no cementite



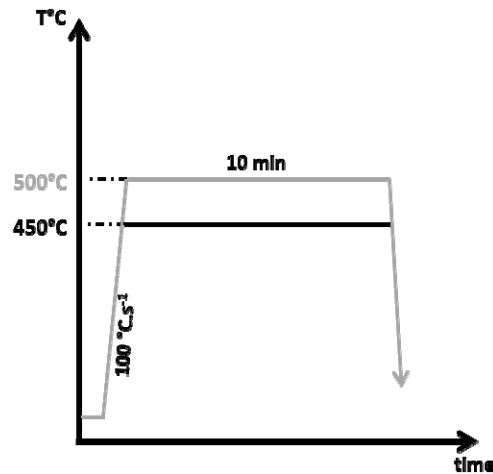
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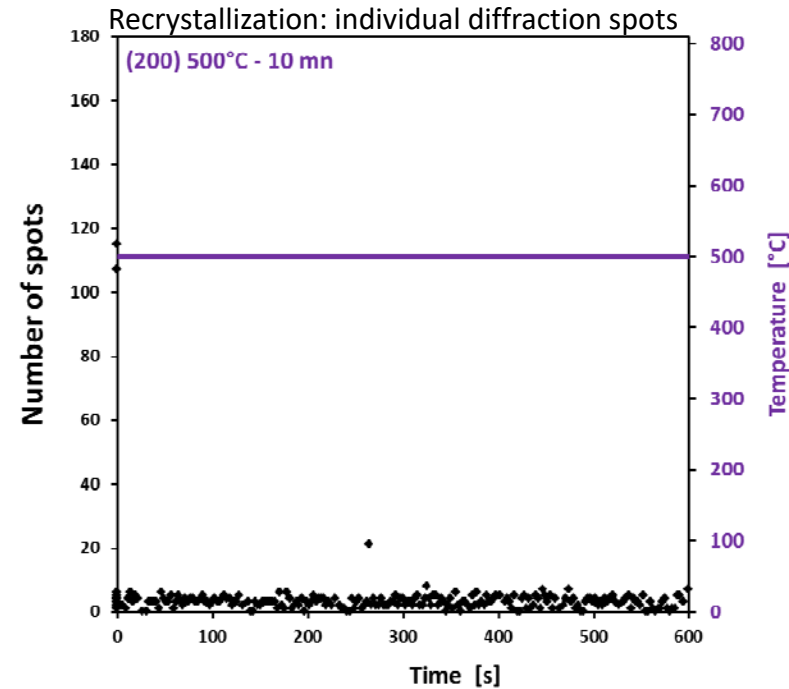
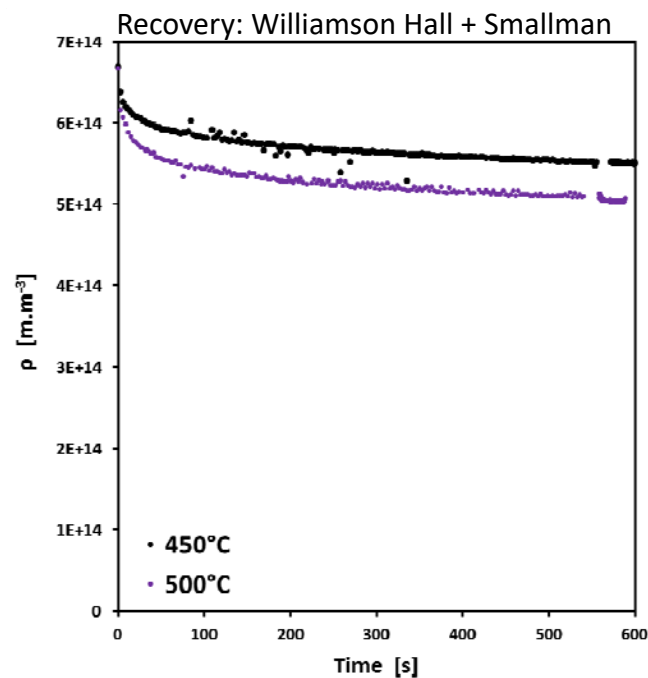
- **450 °C:**
 - Initial ρ_0 : $6.5 \cdot 10^{-14} \text{ m}^{-2}$
 - Logarithmic decrease of ρ
 - No supplementary diffraction spots



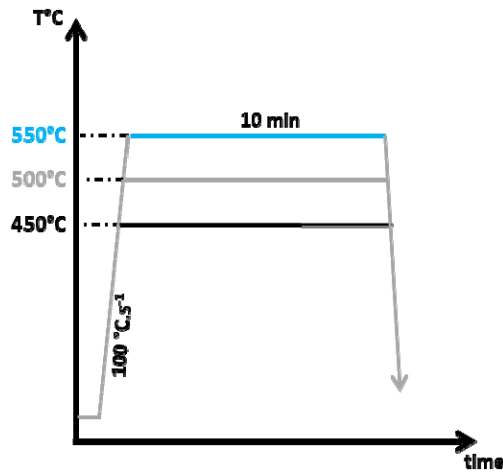
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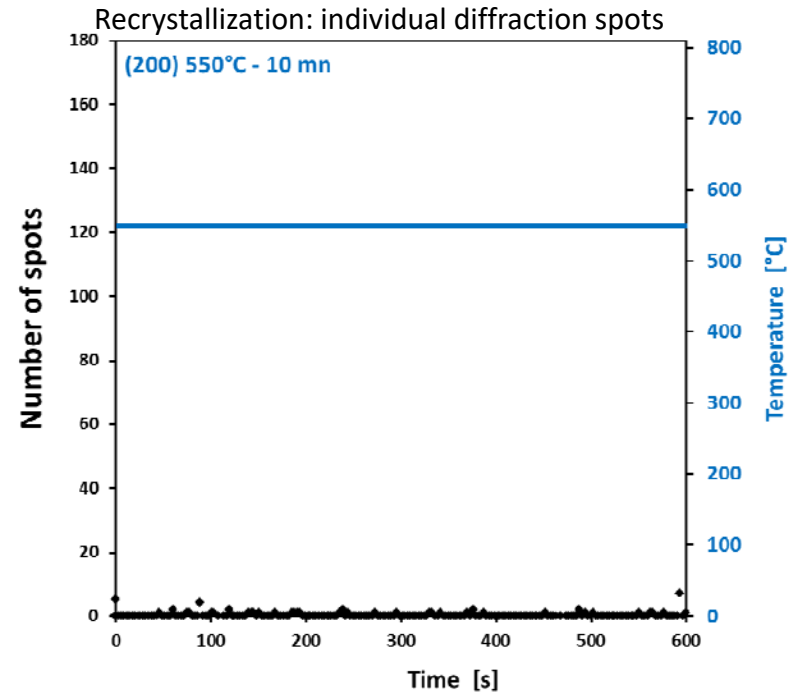
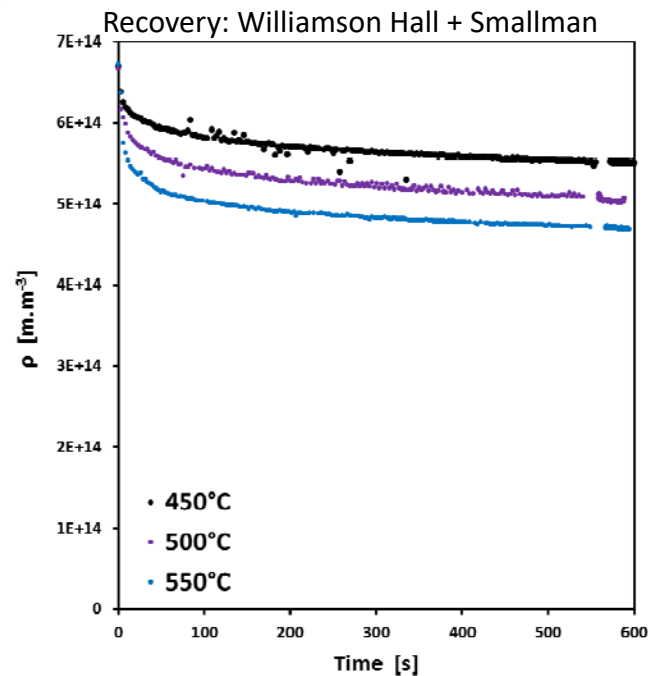
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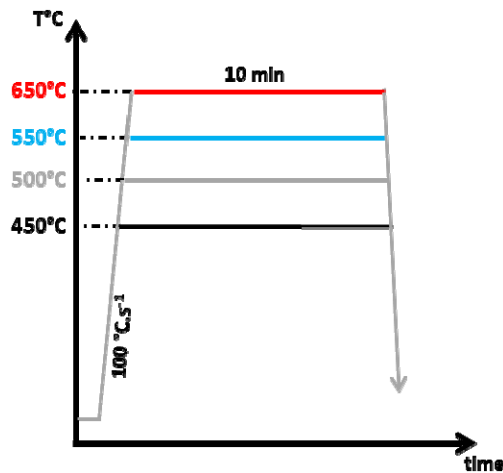
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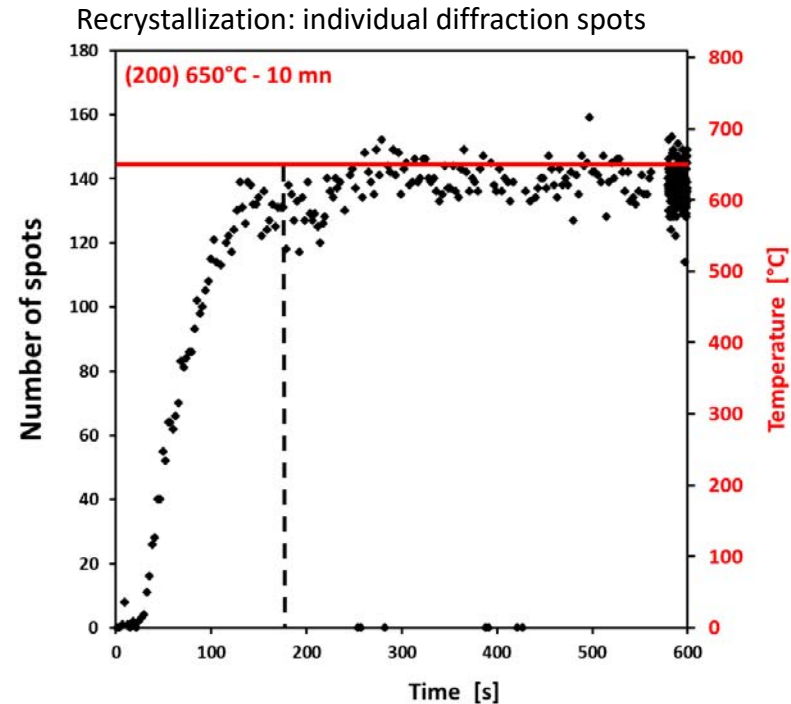
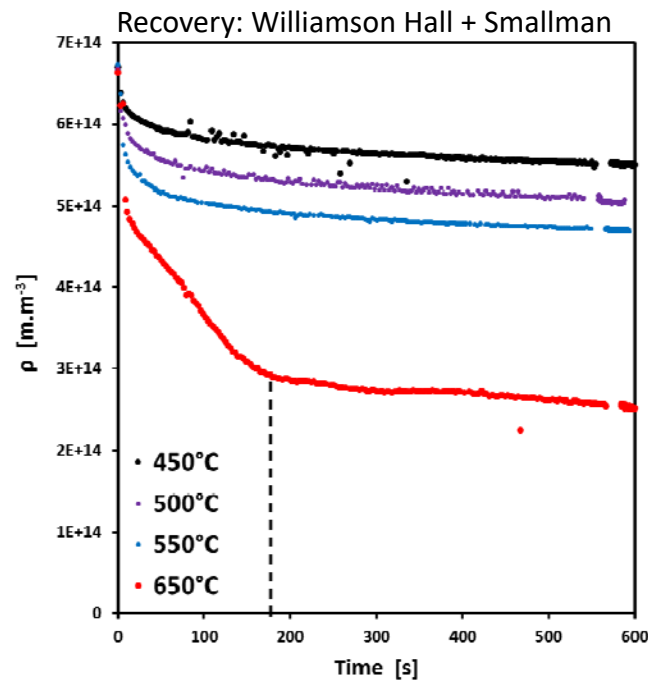
- **550°C:**
 - Logarithmic decrease of ρ
 - No supplementary diffraction spots
 - Activation Enthalpy for Recovery: $Q^{\text{rec}} = 168 \text{ kJ.mol}^{-1}$



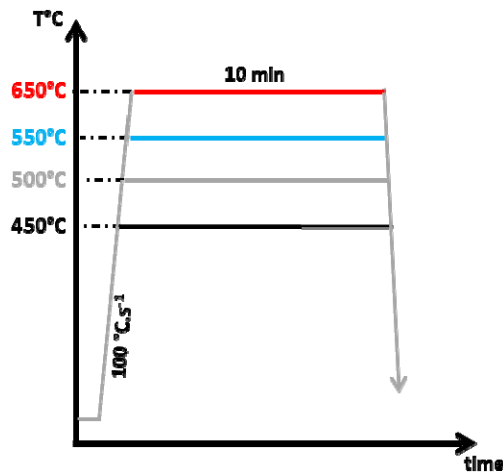
Application on model Fully Ferritic steel



- **650 °C:**
 - Rapid decrease of ρ
 - Followed by linear decrease of ρ
 - Progressive apparition of diffraction spots
 - Short incubation time ~ 15 s

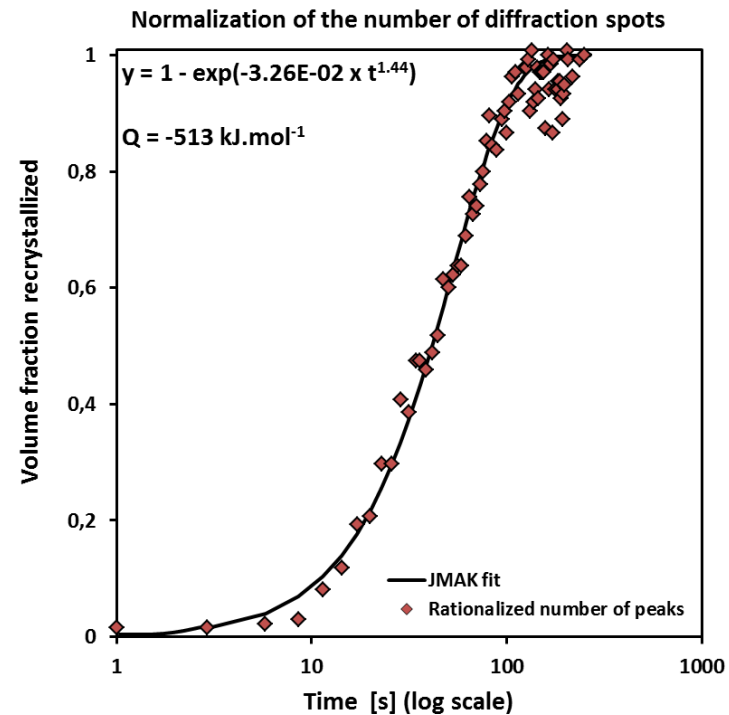
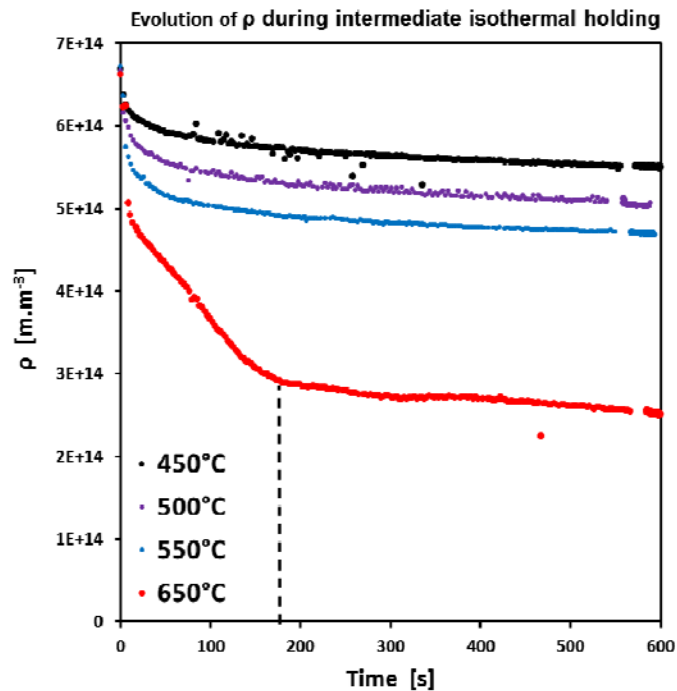


Application on model Fully Ferritic steel



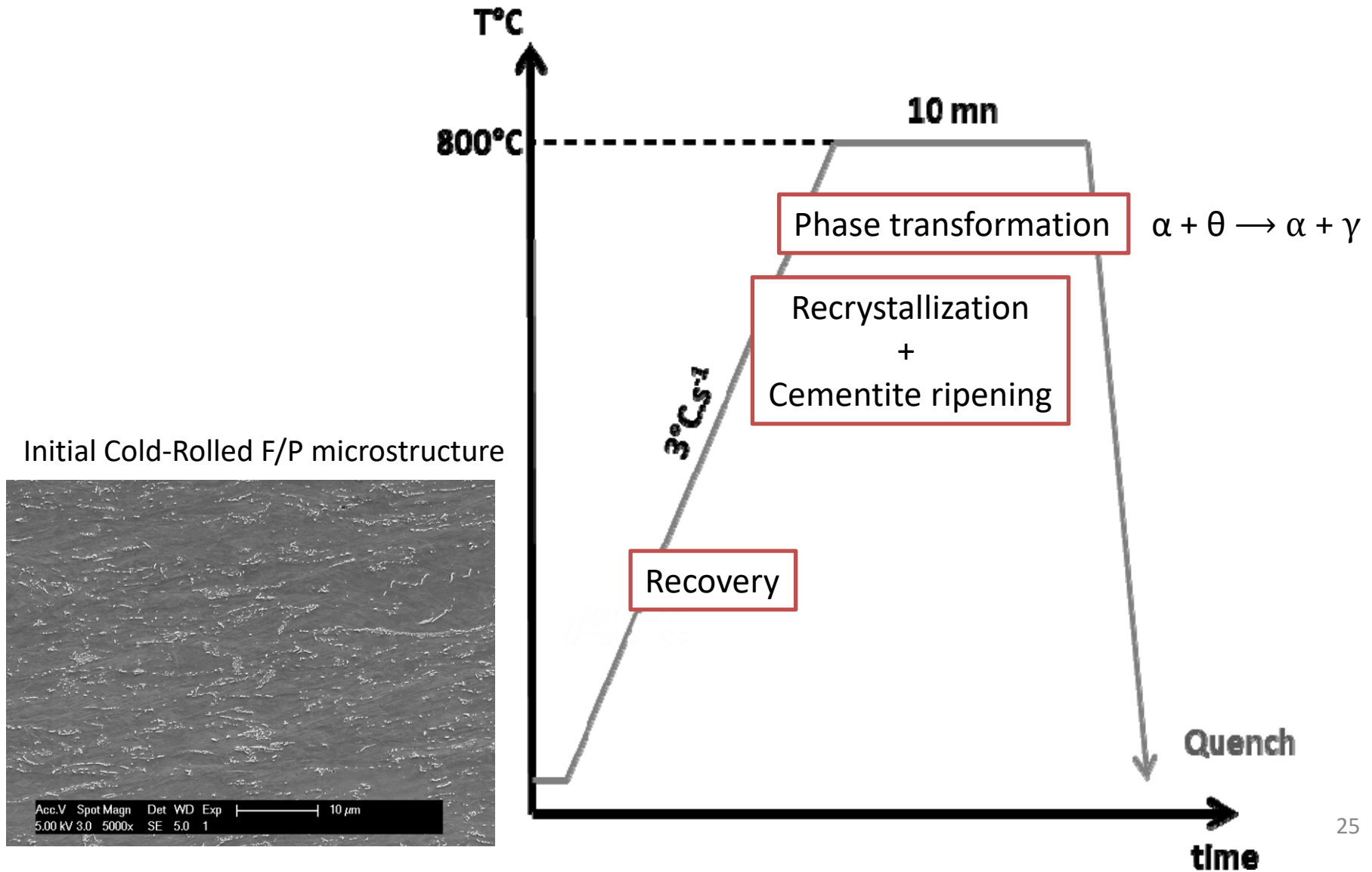
• 650 °C:

- Rapid decrease of ρ
- Followed by linear decrease of ρ
- Progressive apparition of diffraction spots
- Short incubation time $\sim 15\text{ s}$
- Activation Enthalpy calculated: $Q^{\text{rex}} = 513\text{ kJ.mol}^{-1}$, close to 500 kJ.mol^{-1} [MUK96]

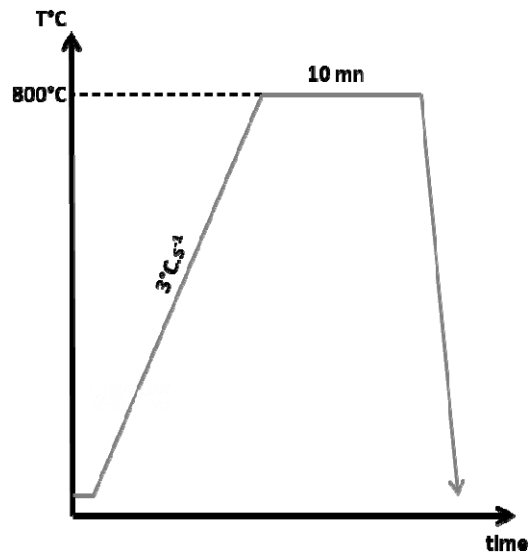


Application on DP Steels

2 additionnal issues: non isothermal treatment + phase transformation



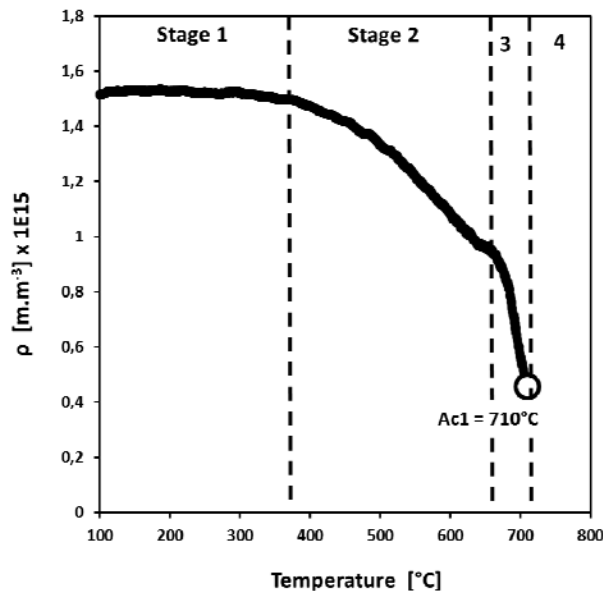
Identification of mechanisms during heating



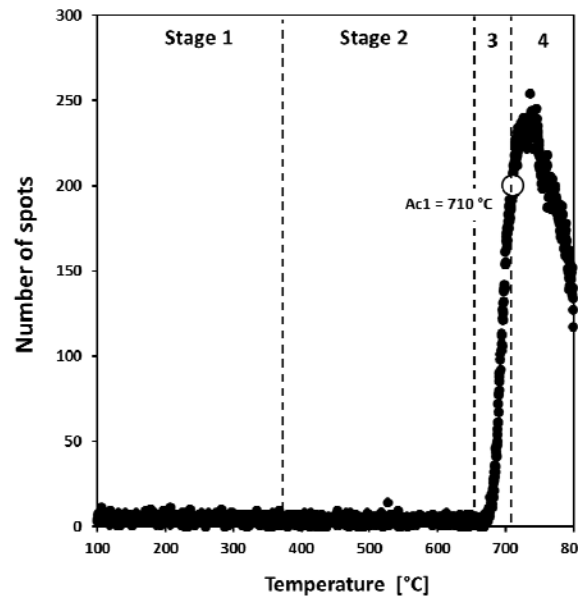
Four stages:

- Stage 1 : no microstructure evolution
- Stage 2 : recovery / no recrystallization
Recovery starts at 380°C
- Stage 3: Recrystallization
– recrystallization starts at 665°C
- Stage 4: Recrystallization + Phase transformation
– The microstructure is ~ 90% recrystallized before austenitization

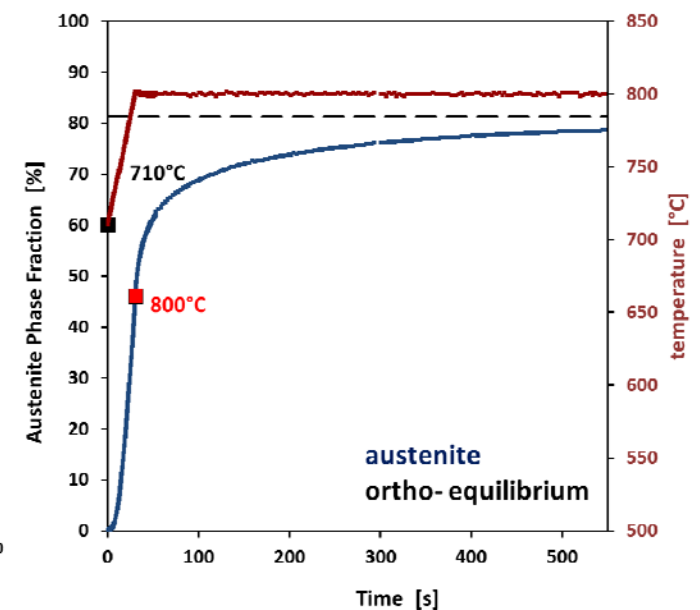
Recovery



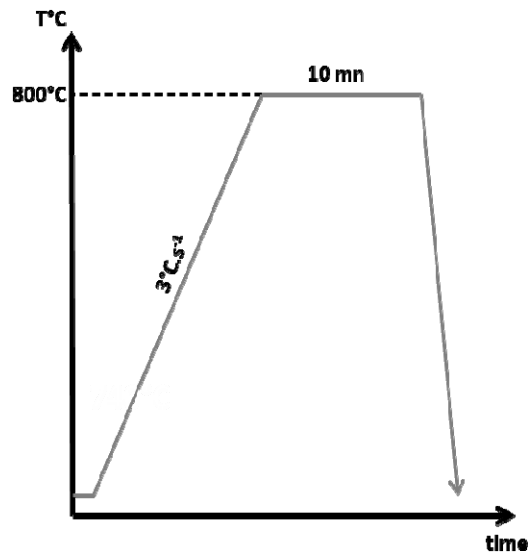
Recrystallization



Phase transformation

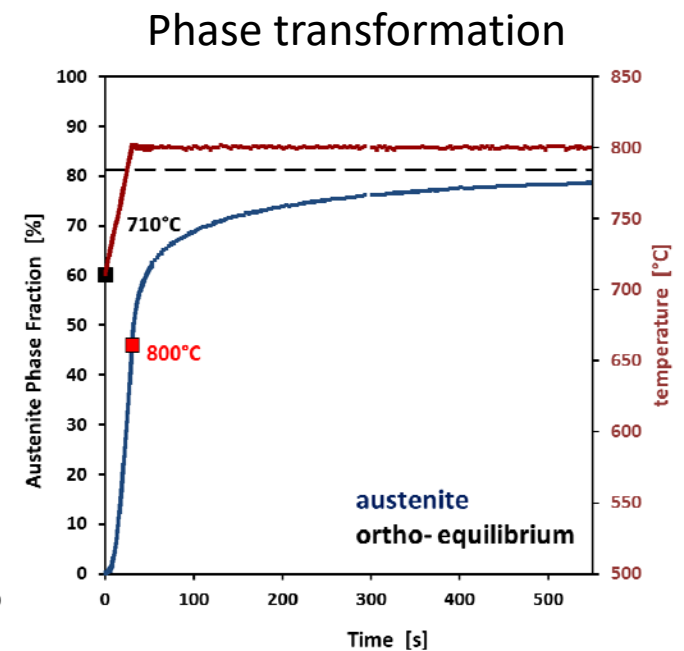
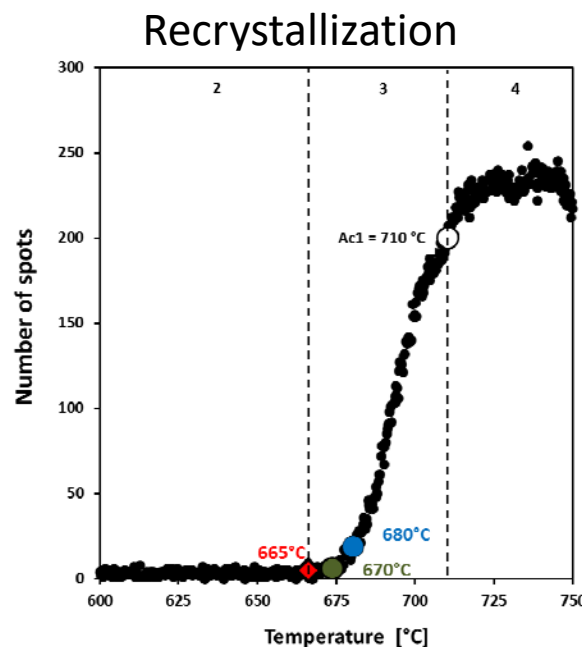
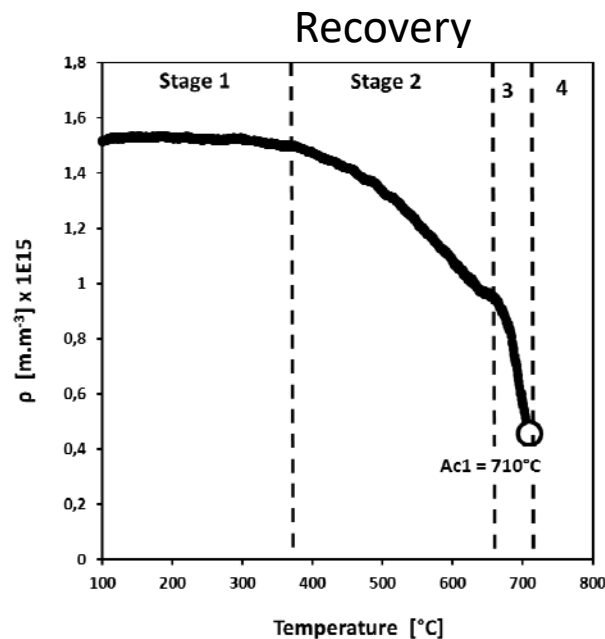


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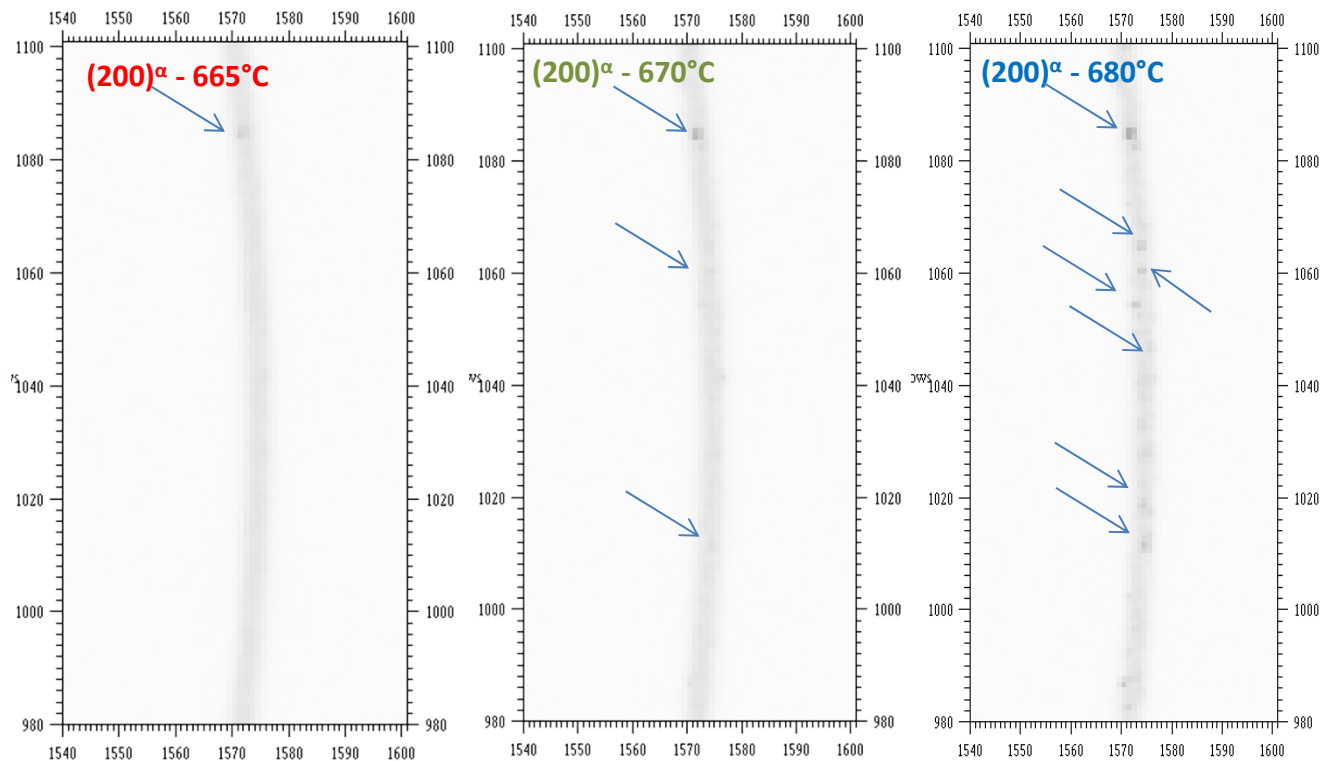
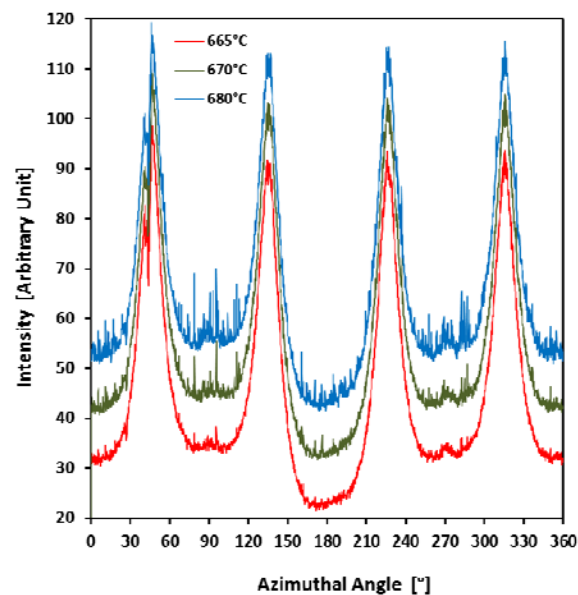
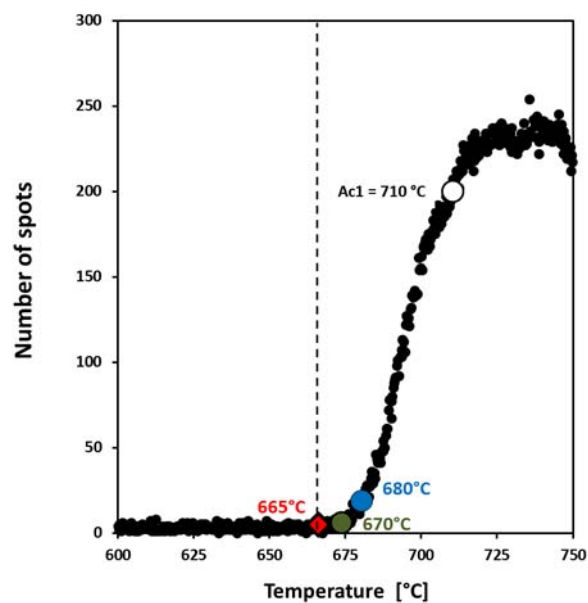


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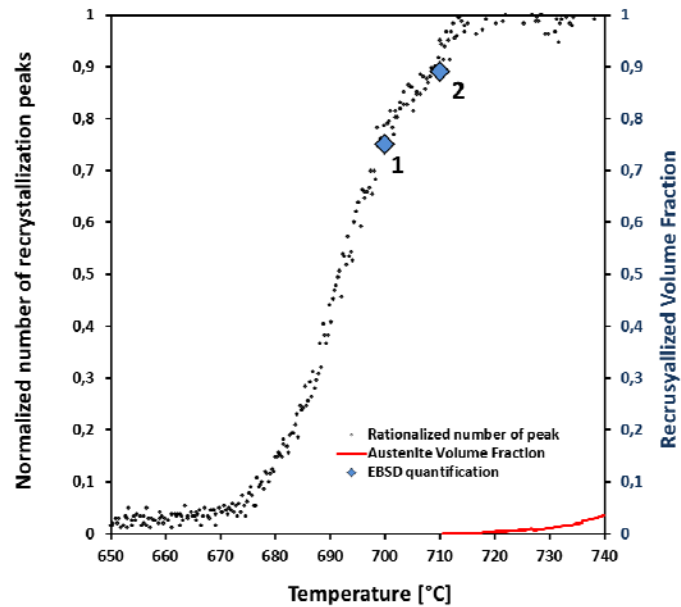


Application on DP Steels

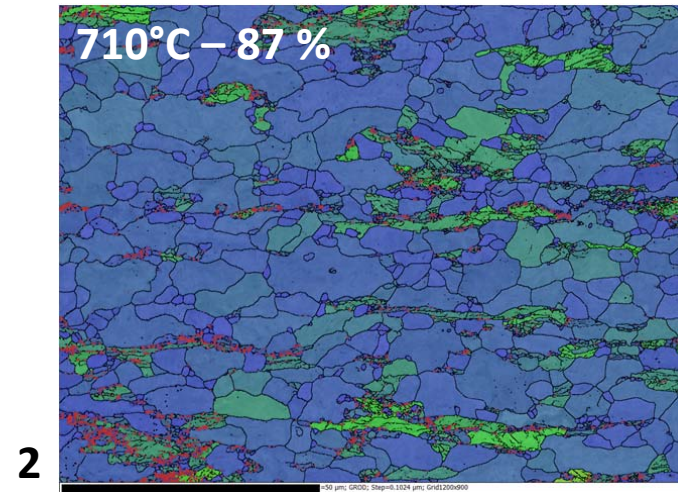
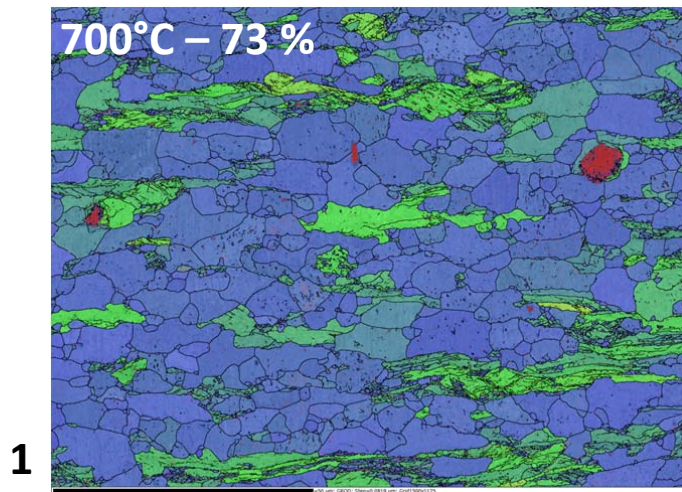


High precision in determining the recrystallization start temperature
Not possible to achieve with post mortem experiments

Application on DP Steels



Comparison with EBSD (GOS):



CONCLUSION

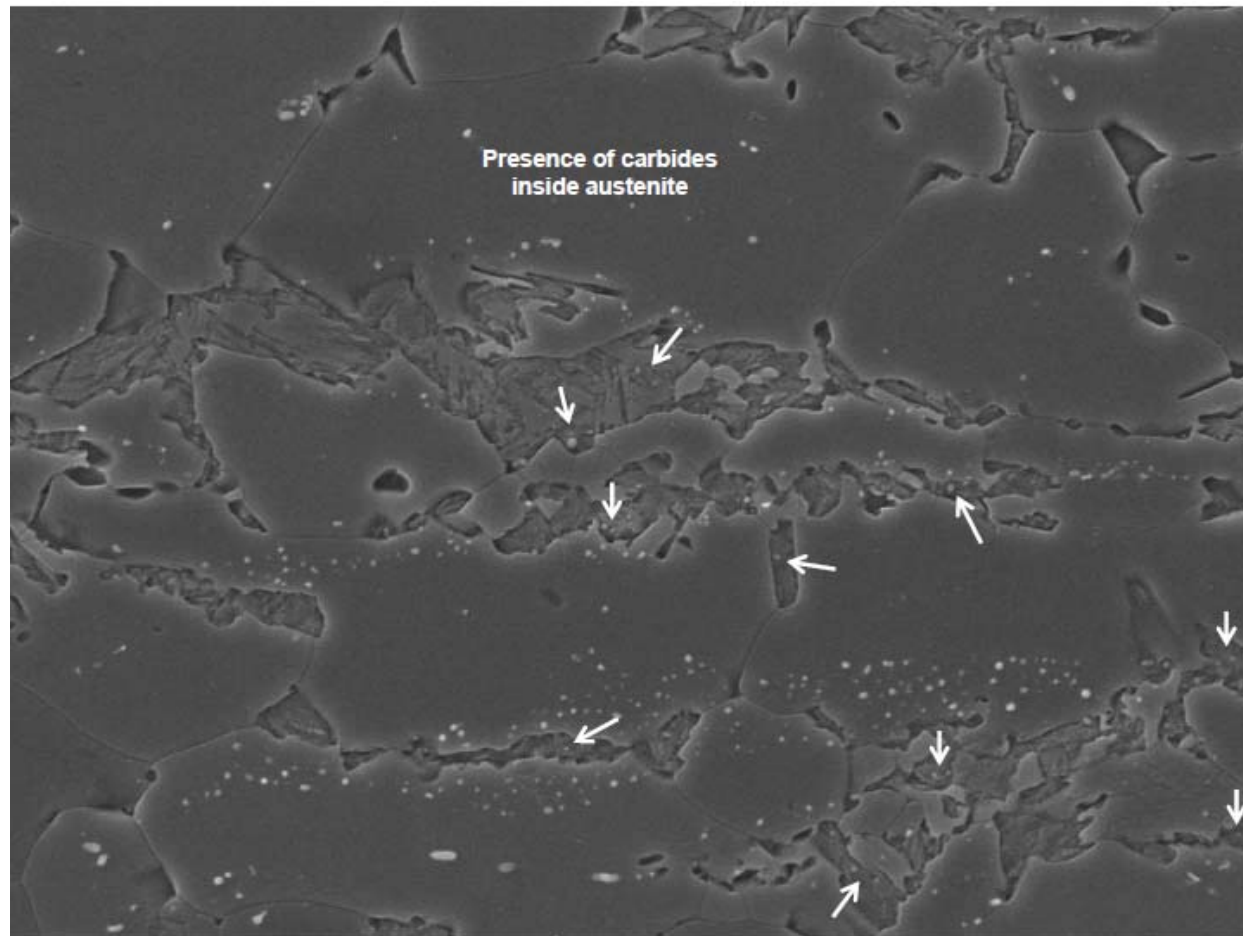
- **HEXRD enables time resolved and *in situ* characterization with a single experiment**
 - Recovery (Williamson Hall + Smallman)
 - Recrystallization (Individual diffraction spots) **NEW !**
 - Phase transformation (Rietveld refinement)
- **Technique tested on:**
 - Ultra Low Carbon (UFC) steel
 - Dual Phase (DP) steel
 - under isothermal / non-isothermal condition

But still need to be validated on more cases

- **Added Values**
 - Precise determination of recovery / recrystallization start temperature
 - Activation enthalpies for recovery and recrystallization

In parallel...

- Modeling of the austenite transformation for:
 - Pearlite islands
 - Dispersed cementite particles



Thank you for your attention

marc.moreno@univ-lorraine.fr