Fédération de Recherche Grand Est Mécanique des Matériaux (GE@2M)

Séminaire 13 Decembre 2021

About the use of Phase Field and FE to predict micostructures, *Application on AlSi10Mg samples produced by Additive Manufacturing*

S. Fetni, J. Delahaye, A. Mertens, A.M. Habraken

LIÈGE université

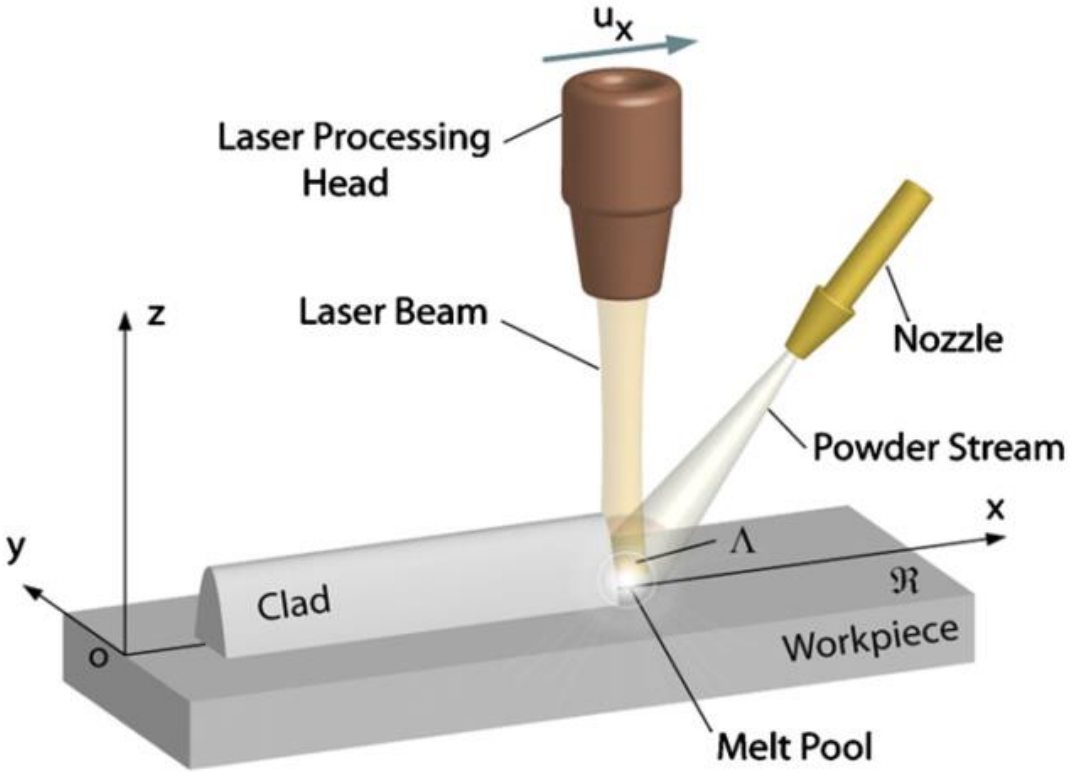
Aerospace & Mechanical

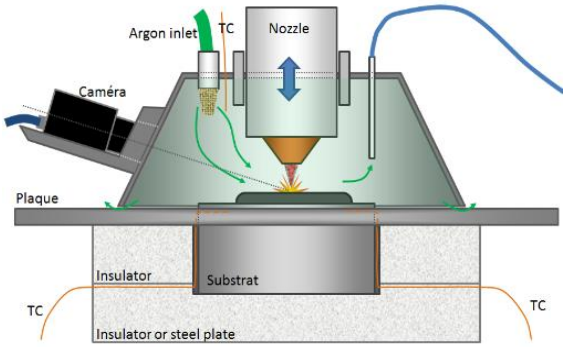
M.M.S Metallic Materials Science

FACULTÉ DES SCIENCES APPLIQUÉES
UEE
Urban and Environmental Engineering

MSM

Laser cladding or Directed Energy Deposition DED

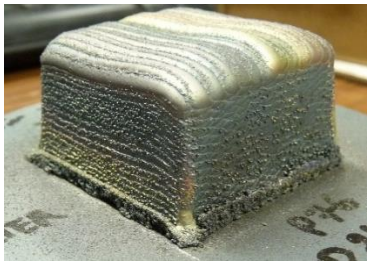




Directed Energy Deposition

Studied Variant:
Powder stream and laser beam are confocal
High power > 1000W
Thick layer 0.X mm

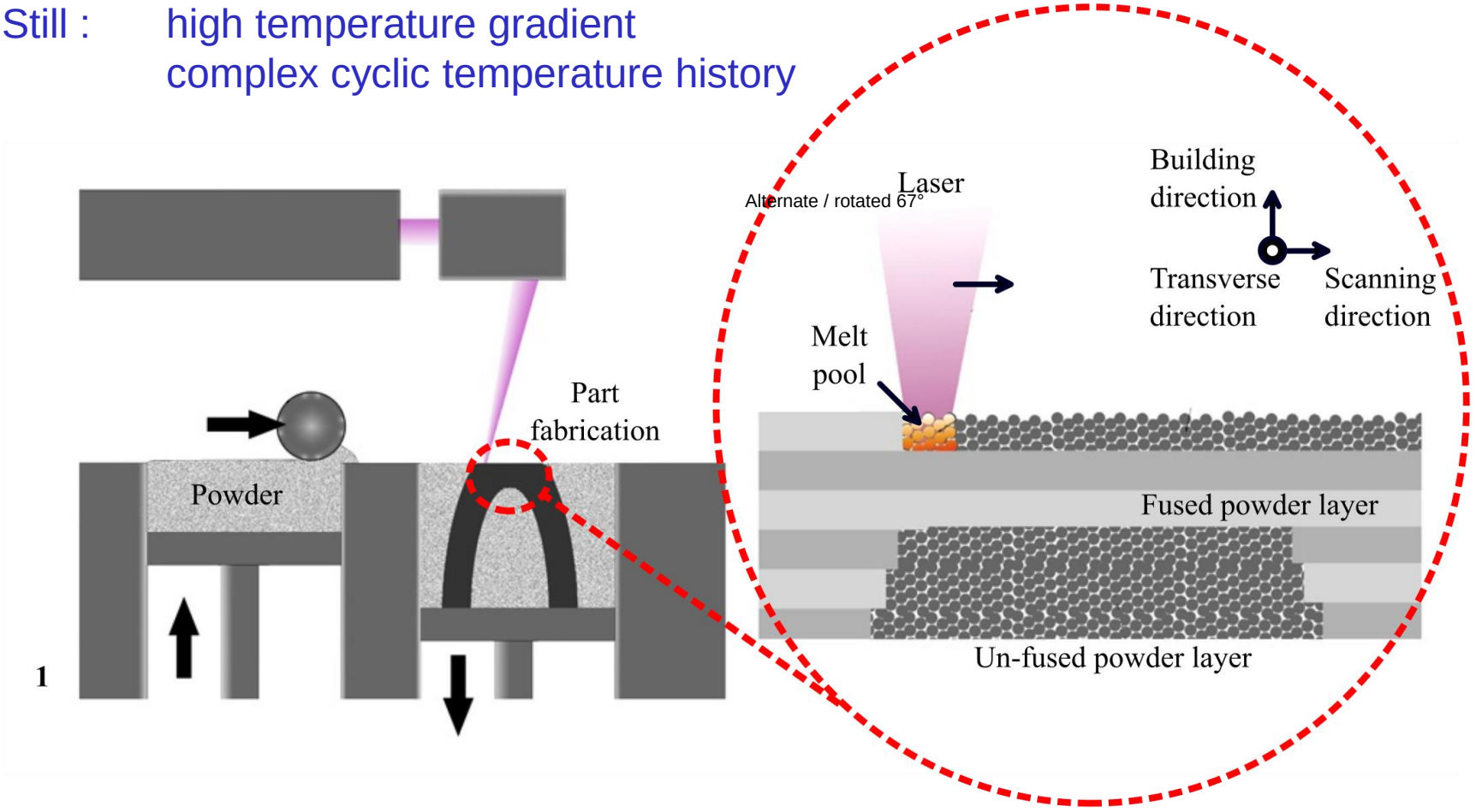
High temperature gradient
Complex cyclic temperature history



Heterogeneity of microstructure formed as previous deposit is heated by the laser high energy

Laser Powder Bed Fusion Process L-PBF (Selective Laser Melting)

Compared with DED : smaller laser energy
Microstructure heterogeneity formed versus height ↓
Job length can generate heat post treated microstructure
Smaller layer deposition per layer
Still : high temperature gradient
complex cyclic temperature history

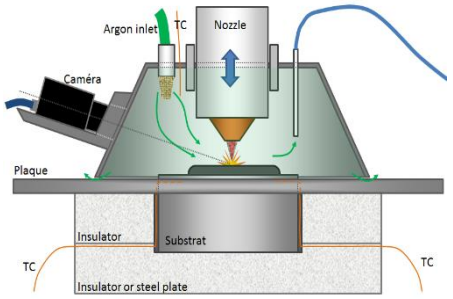


[1] https://en.wikipedia.org/wiki/Selective_laser_melting

Contents

Start with **DED** – "thick" layer but heterogeneity

Accurate thermo-physical properties + double, triple validation(s) of FE results

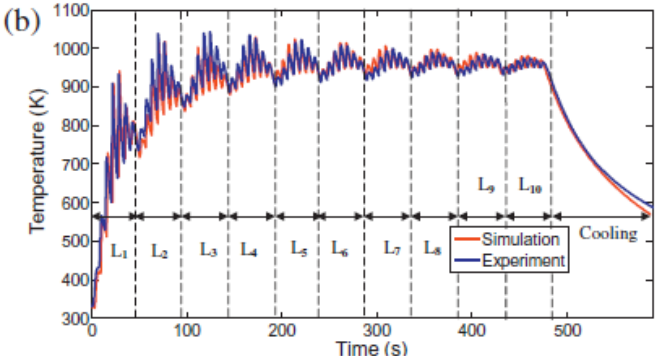


Directed Energy Deposition

-**3D FE models:** each Layer-Track modeled (5 mm height)

- To adjust laser absorptivity & boundary conditions (convection, radiation), multiplicative factor of liquid conductivity

-Ti_6Al_4V alloy: comparison of different laser paths & FE validations



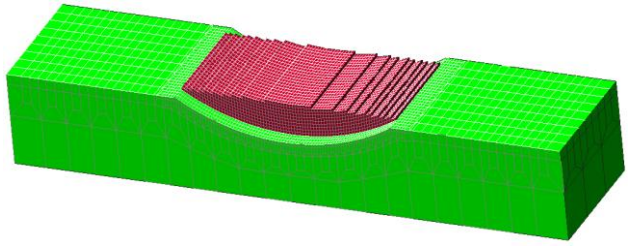
For CTL

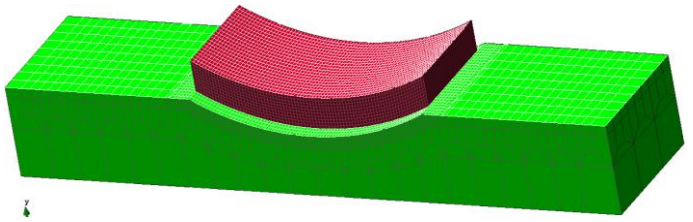
→ measured Tp histories,

→ hardness map

→ Optical or SEM characterization, phase + melt pool shape

Decreased Track Length





Constant Track Length CTL

H.S. Tran Materials and Design (2017), 128

J. Tchundjang *Materials* (2021), 14(11), 2985

Need of accurate thermo-physical properties + double, triple validation of FE results

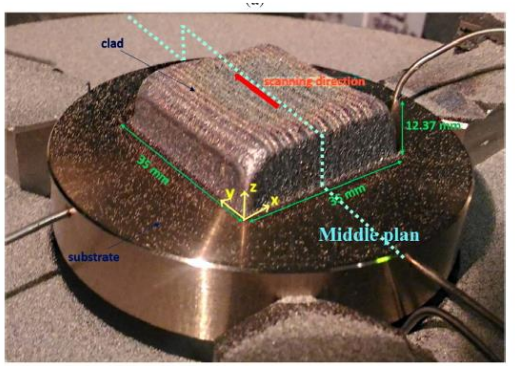
-2D FE models each layer is modeled

to adjust: laser absorptivity (virtual factor)

& boundary conditions (convection, radiation)

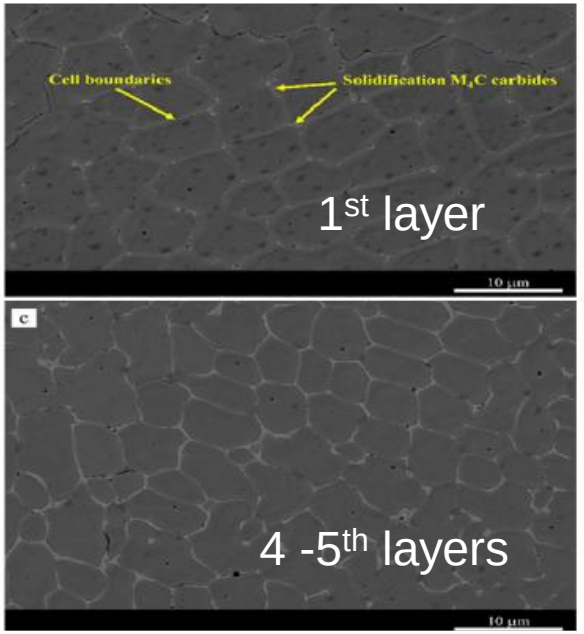
& multiplicative factor of liquid conductivity

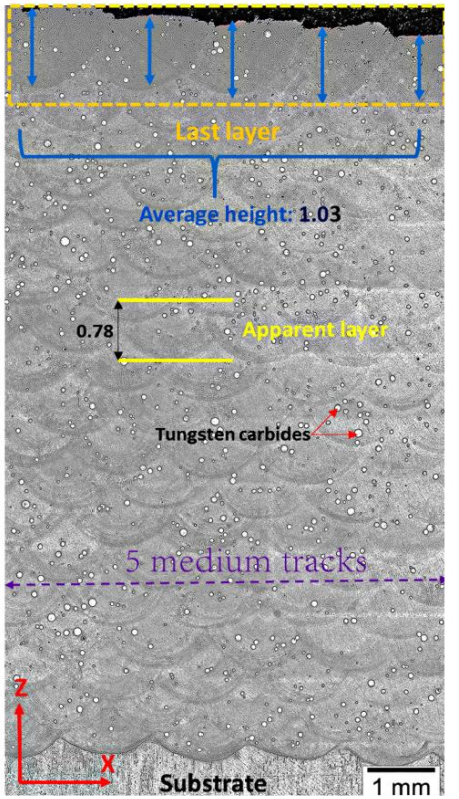
& virtual iddle time



316L + WC: microstructure understanding & FE validations

→ measured Tp histories,
→ melt pool size (OM)
→ carbide distribution (SEM),
→ HAZ (SEM),





S. Fetni Materials & Design (2021), 204(109661)
T. Maurizi-Enciri Advanced Engineering Materials (2020) + submitted (2021)

Need of accurate thermo-physical properties + double, triple validation of FE results

-M4 material (2D or 3D FE models), comparison of bulk or thin wall samples

→measured Tp history,

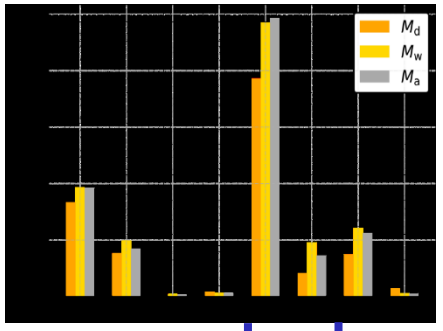
→Optical microscopy: melt pool shape

→SEM : carbide heterogeneous distribution

M4 = high speed steel

-Surrogate model (FFNN FeedForward Neural Network) for uncertainty propagation on melt pool depth, width, area for bulk samples

Sobol Indices



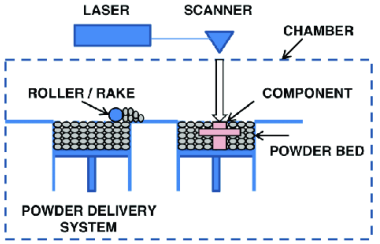
Process parameters

Monte Carlo simulation
FE 25 days… // FFNN 3h30

R. Jardin 2019 Materials Letters, 2020 Metals
T.Q.D. Pham 2021 J. of Intelligent Manufacturing
T.Q.D. Pham Submitted Probabilistic Eng. Mechanics

According *J. Manuf. Sci. Eng*. Apr 2019, 141(4): 040801 conductivity affects L-PBF part quality too….

L-PBF

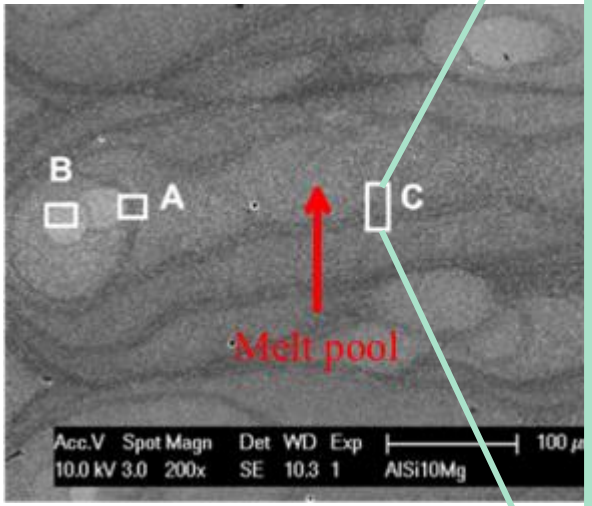


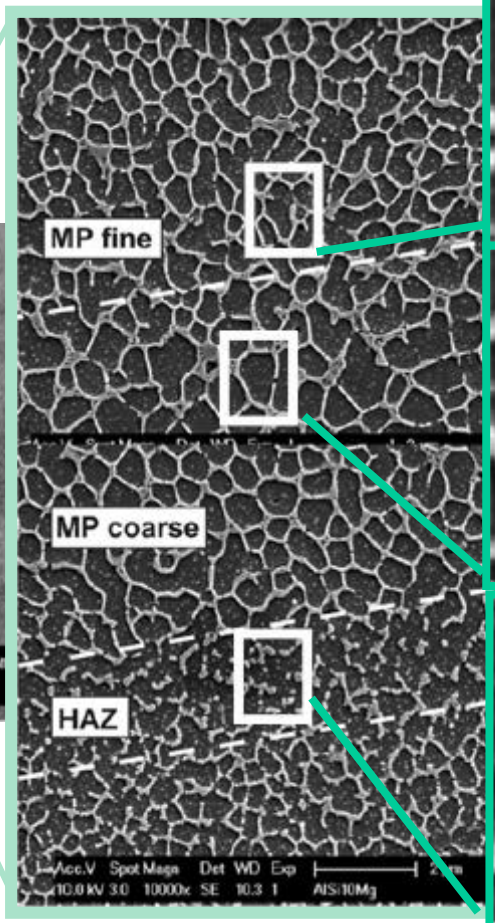
AlSi10Mg Microstructure As Built L-PBF

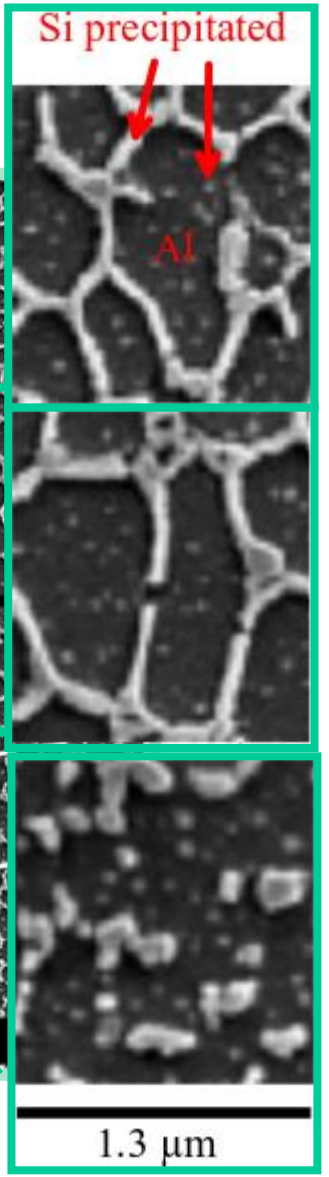
Melt pool of stable size
considering the laser path
bar samples, 60 µm thick layer

Melt pool Core
= fine cell (MPF)

Melt pool Boundary
= coarse cell (MPC)
= Heat Affected Zone (HAZ)







Out of Balance Microstructure

J. Delahaye, Acta Mater. 175 (2019) 160–170.
L. ThijsActa Mater. 61 (2013) 1809–1819.

P = 175W, Alternate / rotated 67°

AlSi10Mg Microstructure As Built L-PBF

Si atoms

→Walls (eutectic rich zone Al Si + Si precipitate)

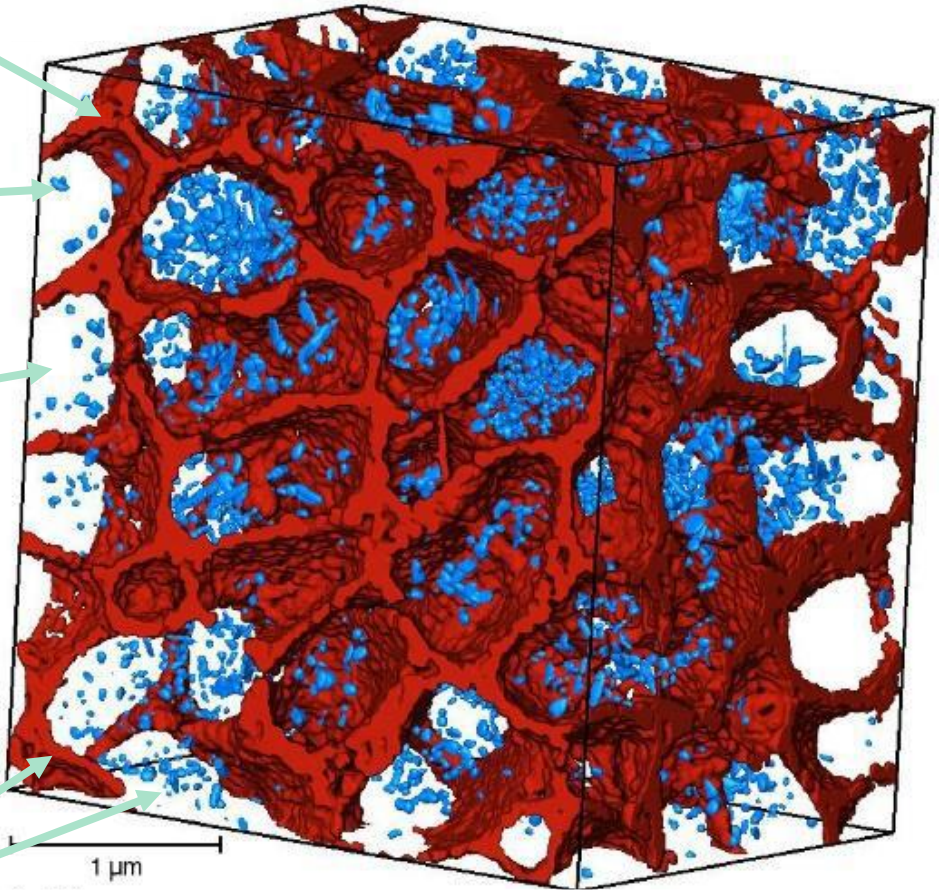
→ Si Precipitate in the cell

→Si in sursaturated* solid solution within the cell

Al atoms

Walls (eutectic rich zone Al Si)

Solid Solution Al Si



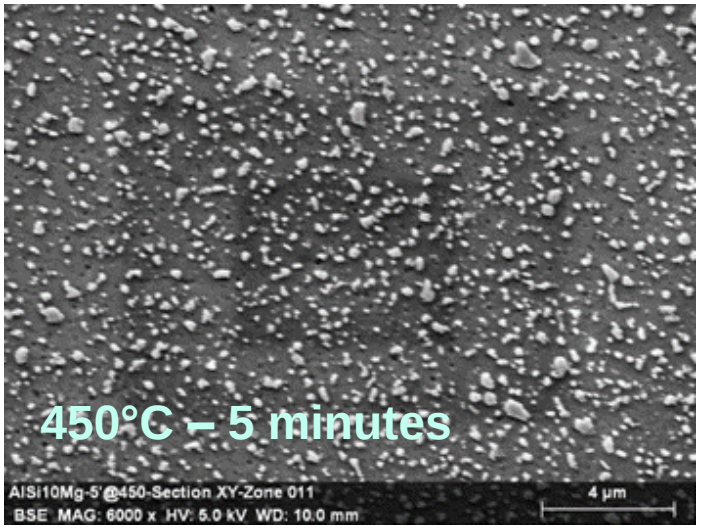
J.G. Santos Macías Acta Materialia 201 (2020)
L. Zhao Materials Science & Engineering A 764 (2019)

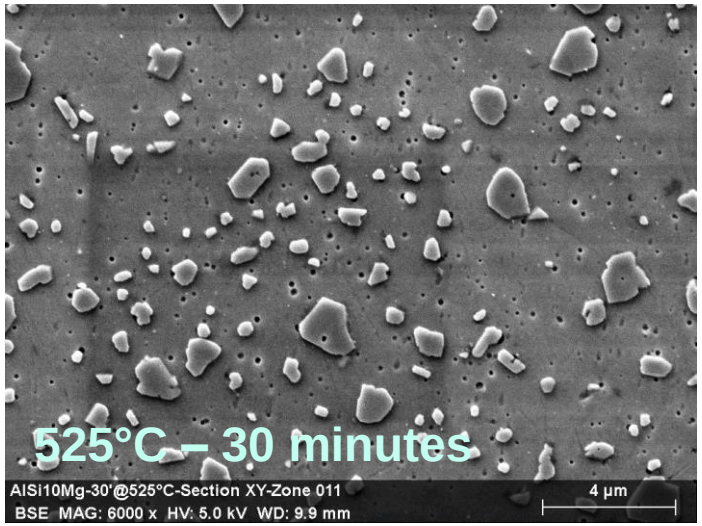
J. Delahaye Acta Materialia 175 (2019)

Plate samples, P 390 W, Thickness layer 30µm but still this skeleton microstructure around cells

AlSi10Mg Microstructure evolution

Thermal treatments applied on As built samples





During L- PBF

in HAZ phase,

“similar” microstructure appears

in Al-Si cells the size of Si precipitates

can varies with process parameters

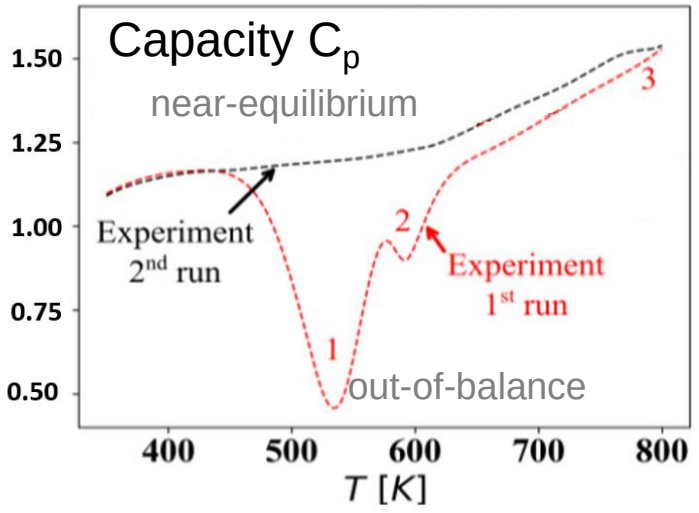
LongLifeAM Wallon Region project results courtesy of MMS team A. Mertens O. Dedry 2021

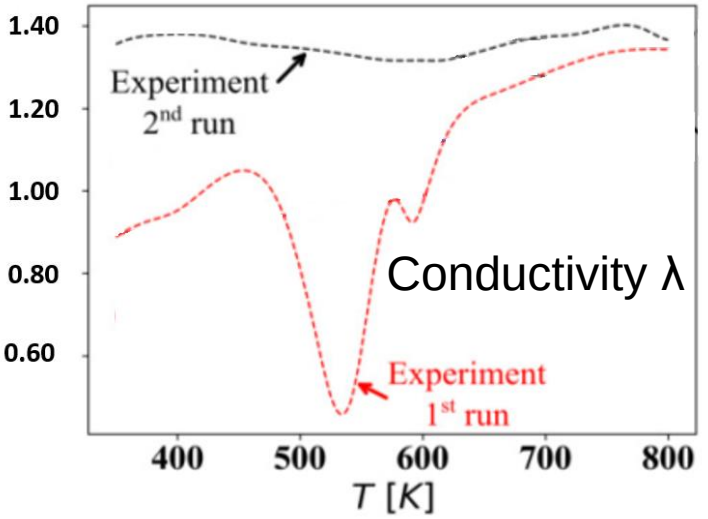
Differential scanning calorimetry (DSC) applied on As-Built sample two times *"1st, 2nd run"* + dilatometry + Laser-Flash of AlSi10Mg

Cooling / Heating rate in DSC:1.7 K.s-1
Direct experimental post treatement
Cooling / Heating rate in L-PBD: 10^6 K.s-1 **What a different range!!!**..

Assumptions*

1. *Si atoms from sur Al-Si saturated solution diffuse to Si precipitates ?*
2. *Si precipitates growth*
3. *Walls dissolve ?*





Phase Field
→ microstructure → analytical formula → C_p, λ

*Y. Birol Journal of Alloys and Compounds. 439 (2007)

Conclusion

FE Model of L-PBF of AlSi10Mg

-needs a data set,

-can provide a Tp° history

Phase-field model of microstructure

-need a Tp° history,

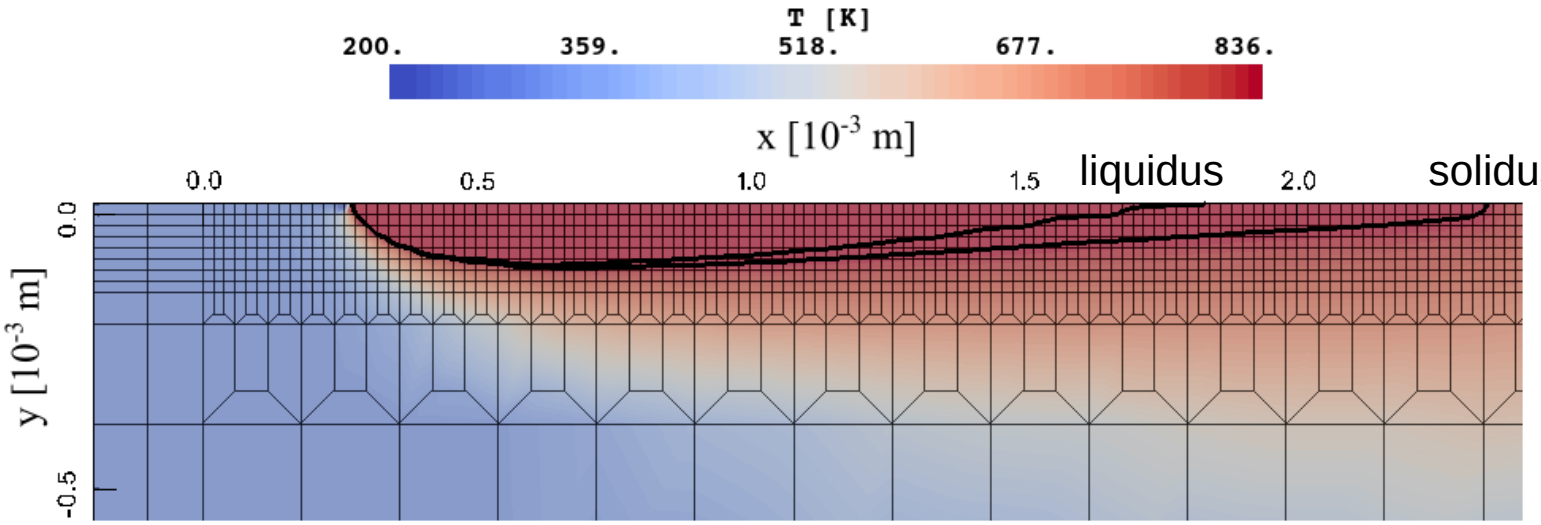
-could provide data set for FE ?

Contents

Laser Power 370 W
Laser velocity 1.3ms^{-1}
Built platform 35°C

FE simulation features

Lagamine non linear software with birth element technique



Just a part, mesh is larger (width 3.64 mm and height 3.79 mm)

-the “best” thermo-physical properties data set (next slide)
& “physical idle” time based on process parameters
& boundary conditions (convection, radiation) based on literature
& **tuned** laser absorption coefficient

-the vertical profile of tp° for different element sizes (10, 20, 40 µm)
→Temperature field is converged, 40 µm is OK

-the case of 5 layers has been studied (remeshing strategy)
→ Temperature history between the different layers are stable

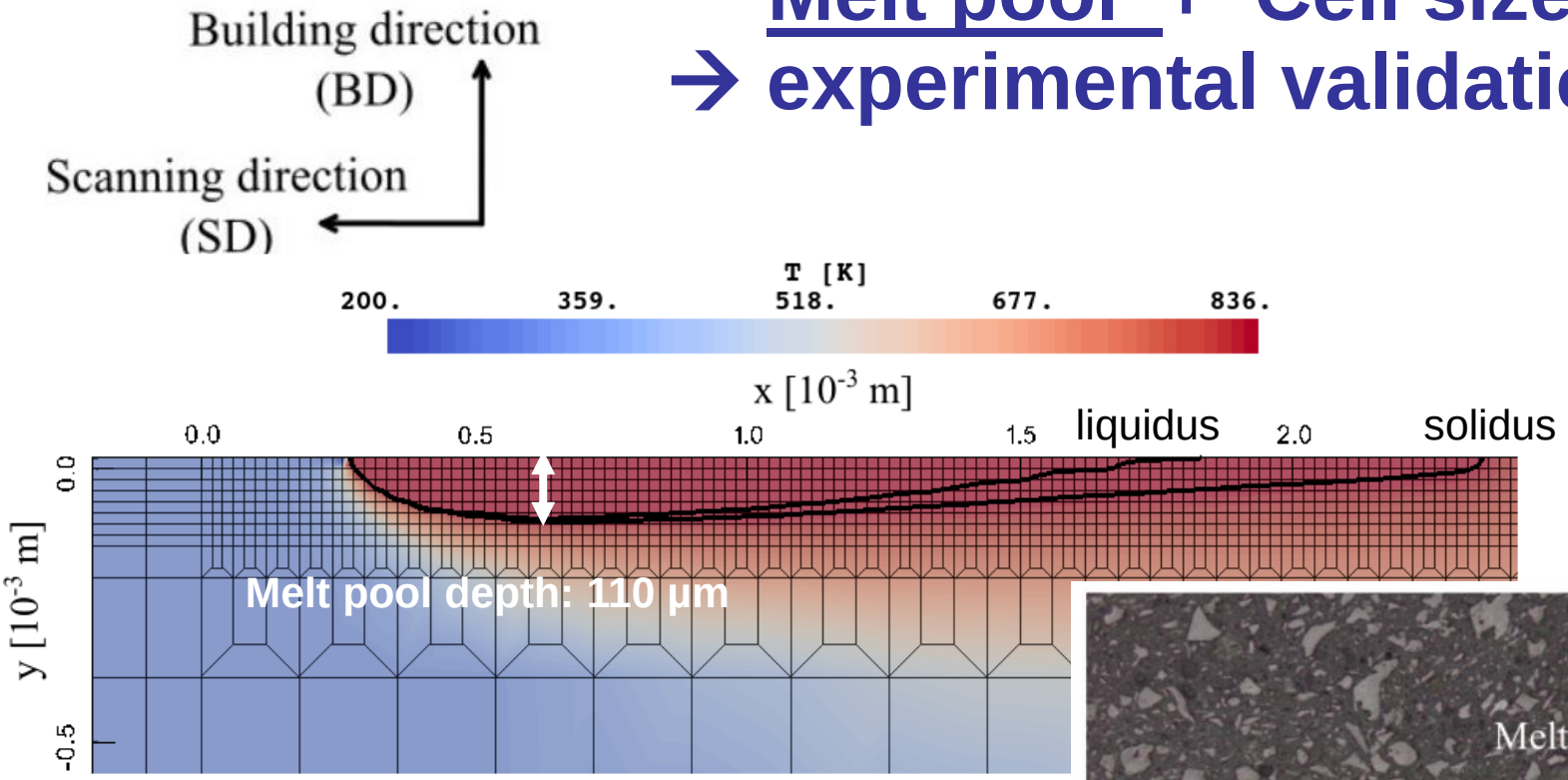
→ Discussion
40 µm element
1 Layer case

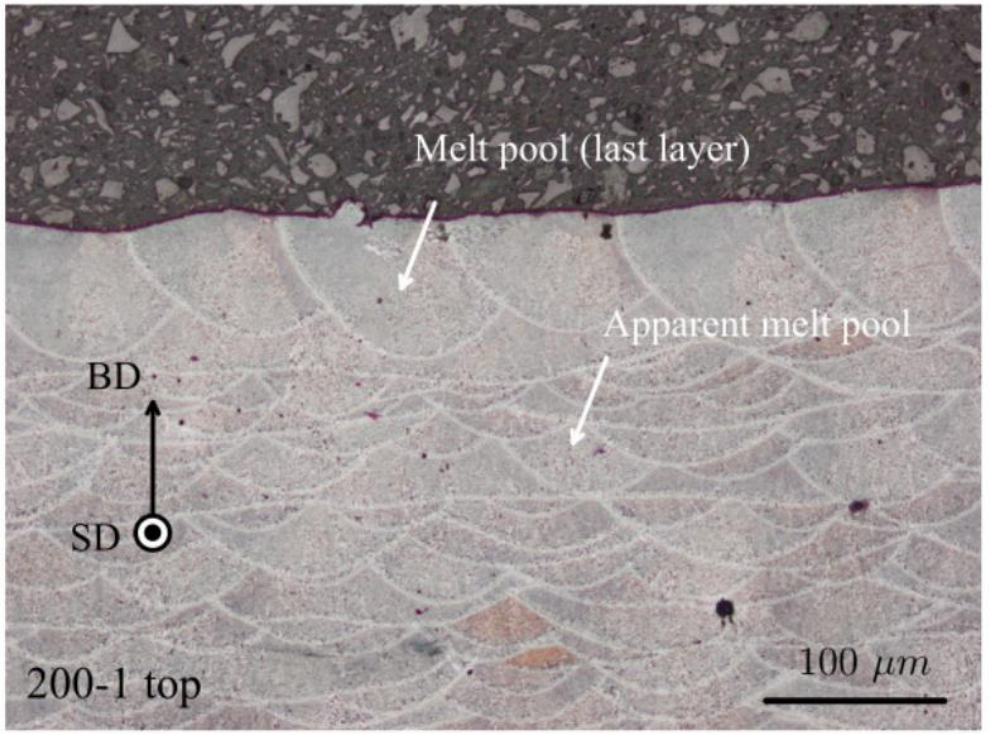
Thermo Physical data set for FE model

- Analytical model **for the powder**: Zener-Schlunder like model
  (Sih and Barlow 2004 Particul. Sc. Technol. 22.4)
- Rapid solidification model** (Al-Si system) **for mushy state** in L-PBF condition
- CALPHAD (Al-Si system) computation at high tp°, different improvements tested
- DSC Measurements **for the metastable As built microstructure** in solid state
- Measured value **for the liquid state** with a factor 1 or 2 for taking into account Marangoni effect
  (liquid convection generated by variable surface tension in the melt pool)

*R. Trivedi Dendritic growth, International Materials Reviews 341 39 (2) (1994)
**w. J. BOETTINGER'Fourth conf. on rapid solidification', 13; 1988, Baton Rouge, LA, Claitor's Publishing Division.

Melt pool + Cell size
→ experimental validation

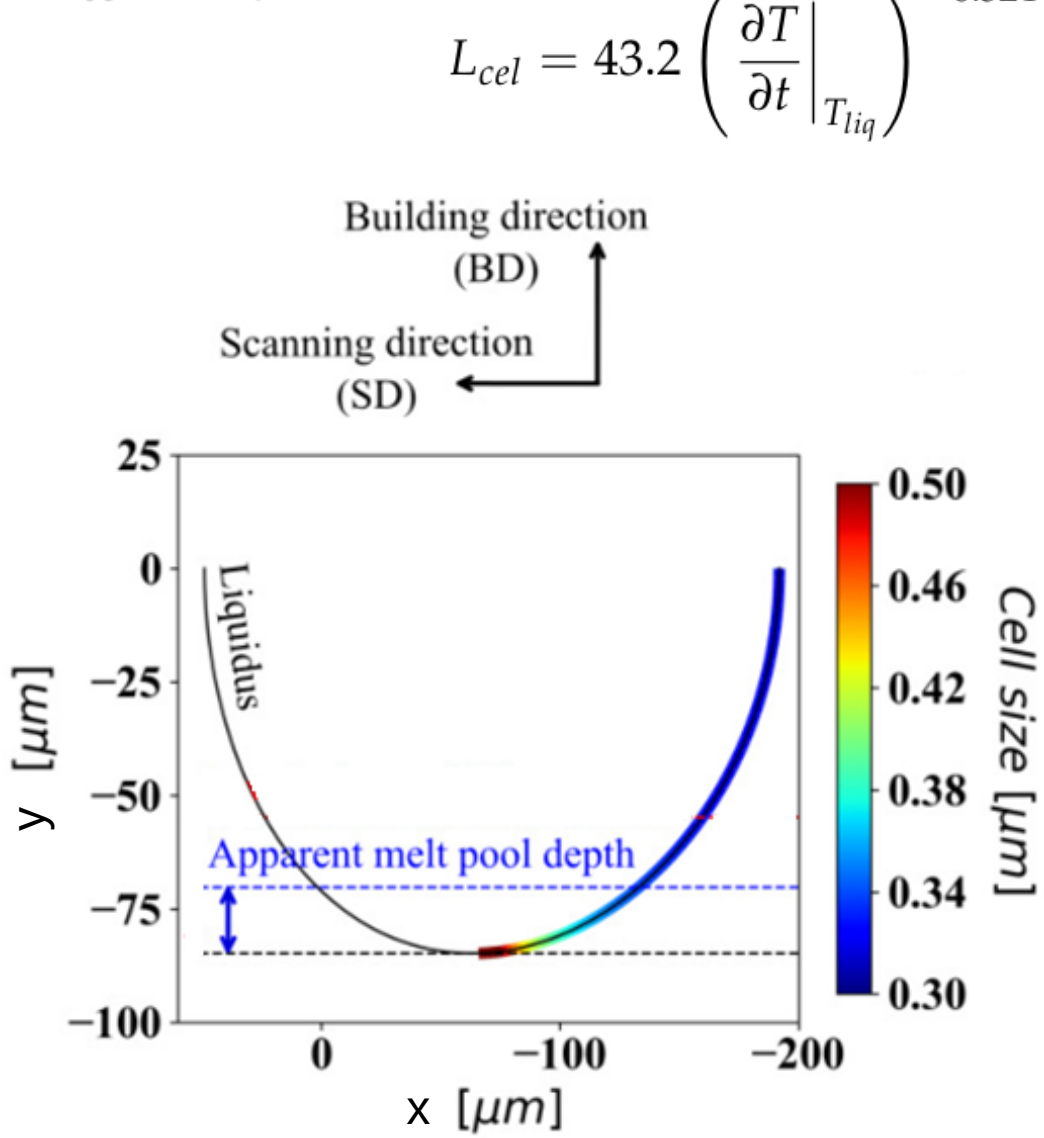


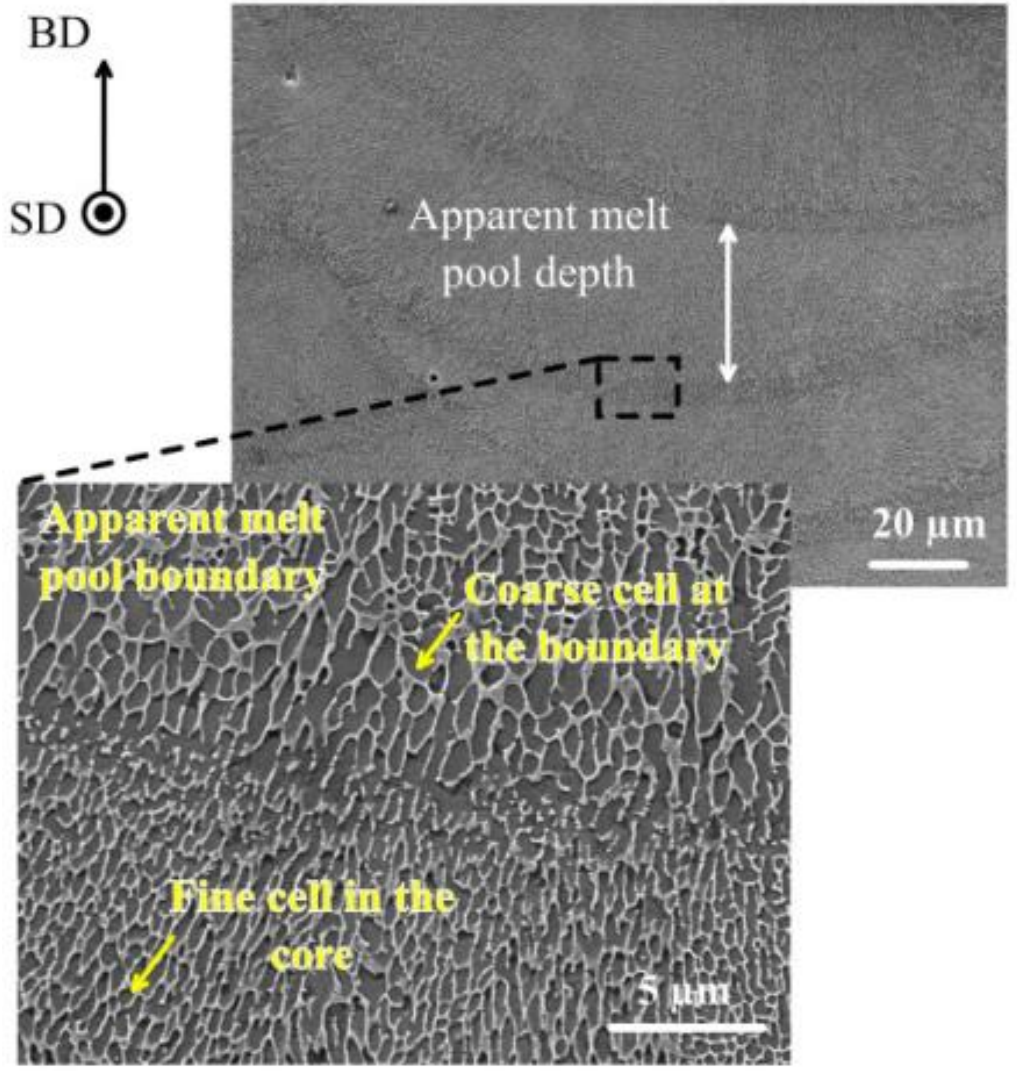


Melt pool and Cell size → experimental validation

Matyja's* equation :

$$L_{cel} = 43.2 \left(\left. \frac{\partial T}{\partial t} \right|_{T_{liq}} \right)^{-0.324}$$



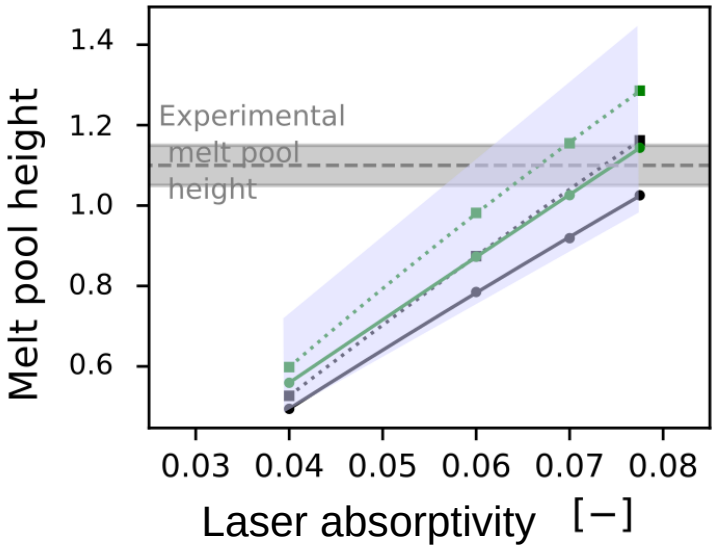


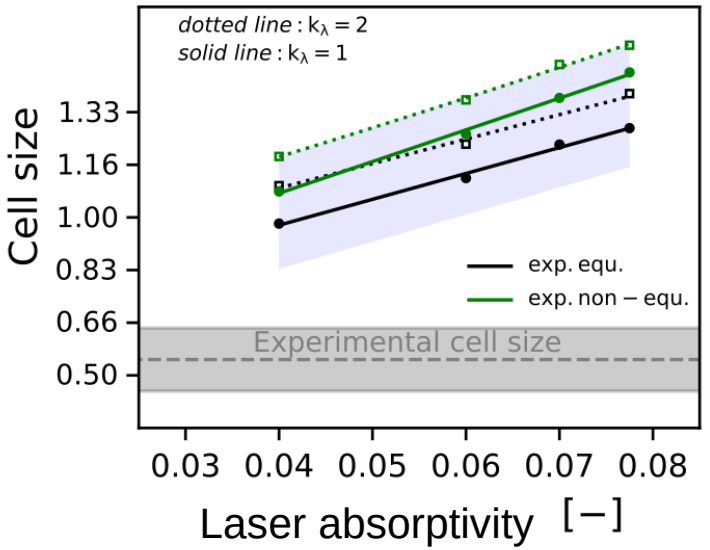
* H. Matyja, Journal of the Institute of Metals. 96 (1968) 30–32.

Tuning laser absorptivity

For fixed convection & radiation coefficient,
identified laser absorptivity highly depends on input data

FE Prediction





FE Assumptions

c_p and k

Shaded zone: Calphad with different assumptions

DSC experiment

1st Run
2nd Run

… Marangoni
k x 2

Why ?

-2D FE assumption
-solid FE model
-Accuracy of material thermo-physical properties

Contents

- ✓ Background & motivations
  - FE model in AM
  - AlSi10Mg Microstructure evolution
- ✓ FE Model of L-PBF of AlSi10Mg :

  needs a data set, can provide a Tp° history
- ❑ Phase-field model of microstructure evolution:

  need a Tp° history, could provide data set for FE ?
  - Model description
  - Model parameters
  - Simulated calorimetric curve

Conclusions

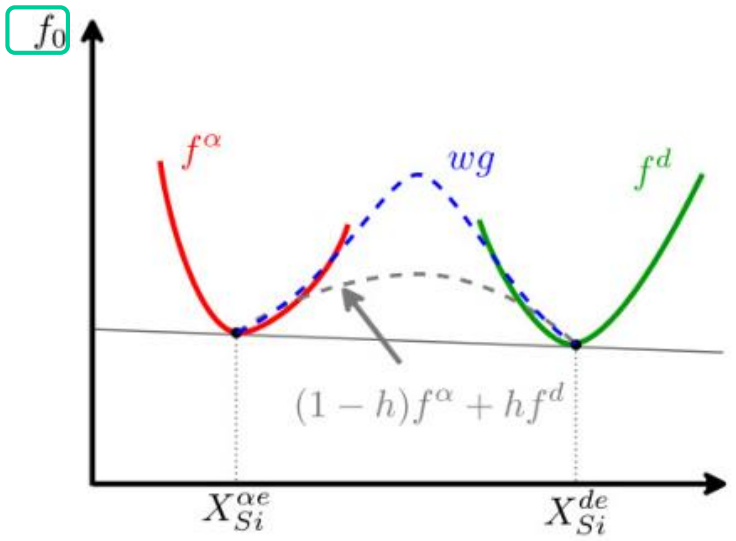
Phase Field Model

Free energy formulation

- Kim Kim Suzuki “KKS” model*
- Interface considered as mixture of both phases with the same chemical potential

Homogeneous free energy density

η=1 Diamond d Si
=0 FCC Al α



e for equilibrium

Molar fraction of Si

Non-homogeneous free energy

Homogeneous free energy density

$$\mathcal{F} = \langle V_m \rangle \int_V \left(f_0(X_{Si}, \eta, T) + \frac{\kappa^2}{2}(\nabla\eta)^2 + e_{el} \right) dV$$

Average molar volume

Molar fraction of Si

Phase

Temperature

Interface contribution

Elastic strain energy

Double-well potential function of height w

Interpolation function

Si precipitate

Al matrix

$$f_0(X_{Si}, \eta, T) = \frac{1}{\langle V_m \rangle}[h(\eta) f^d(X_{Si}, T) + (1 - h(\eta)) f^\alpha(X_{Si}, T) + wg(\eta)]$$

* S.G. Kim Physical Review E 60 (6) (1999) 12 and J.Z. Zhu, Acta Materialia 52 (2004)

Phase Field Model

Elastic strain energy $\Rightarrow$ $e_{el} = \frac{1}{2} C_{ijkl}(\varepsilon_{ij} - \varepsilon^0_{ij})(\varepsilon_{kl} - \varepsilon^0_{kl})$

Stresses

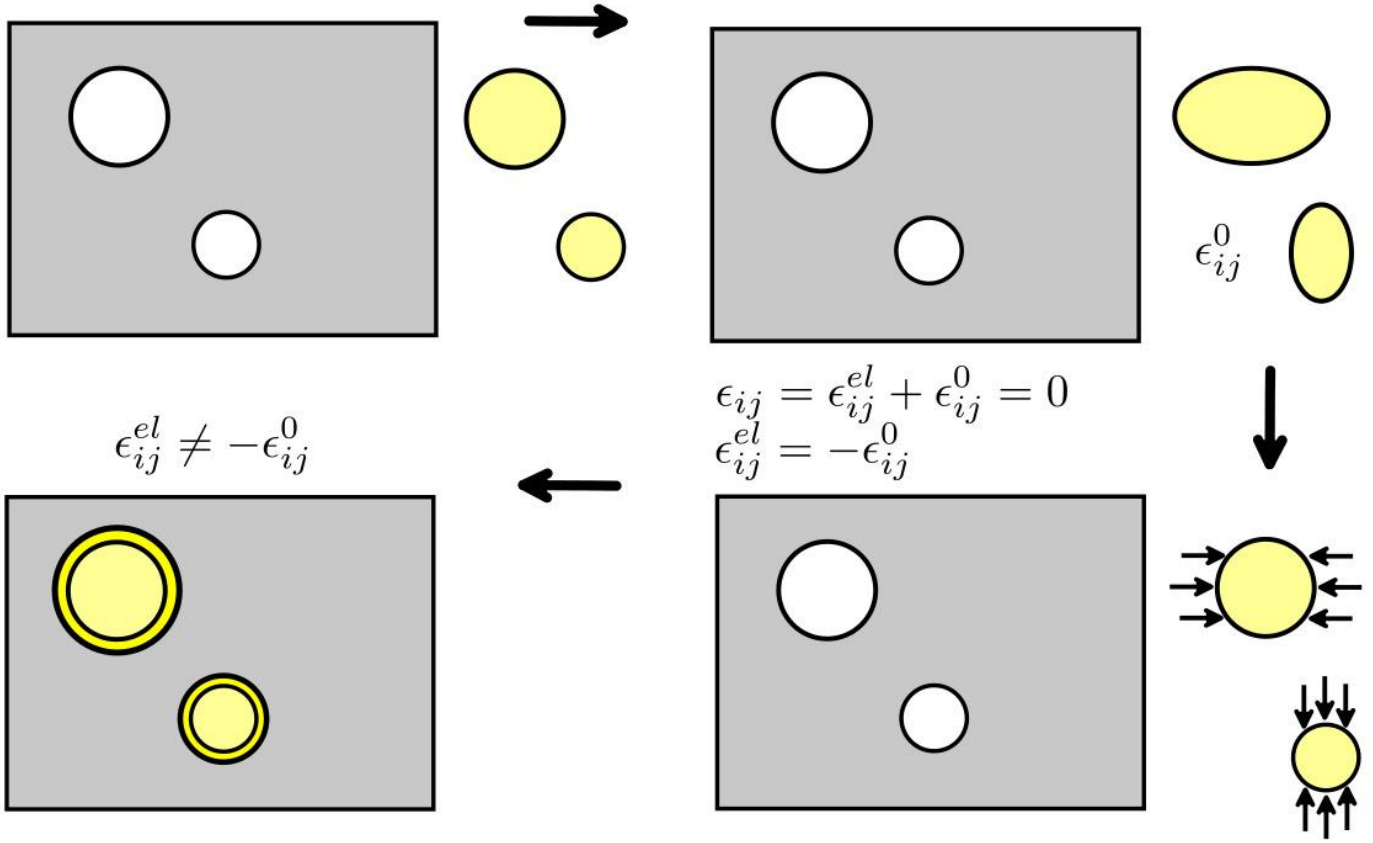
Total strain

Eigen strain

$$\begin{cases} \nabla_j \sigma_{ij} = 0 \\ \sigma_{ij} = C_{ijkl}(\epsilon_{kl} - \epsilon^0_{kl}) \end{cases}$$

Elastic strain

Stiffness tensor



N. Moelans
Calphad 32
(2008) 268–294

*A. Khachaturyan, Theroy of Structural Transformations in Solids, 1983.

Phase Field Model

Excess quenched-in vacancies* present in as built samples of L-PPF

Non-equilibrium vacancy site fraction

$$X_{Va}(t+dt) = X_{Va}(t) + \left[1 - exp\left(-\frac{D_{Va}}{L_{Va}^2}dt\right)\right]\left[X_{Va}^{e}(T(t)) - X_{Va}(t)\right]$$

Mean vacancy diffusion path

Vacancy site fraction at equilibrium

Vacancy diffusion coefficient

Impurity diffusion coefficient of Si in Al

$$D_{Va} = \frac{X_{Va}}{X_{Va}^{e}}\left(X_{Si} * D_{Si}^{Al} + (1 - X_{Si}) * D_{Al}^{Al}\right)$$

Molar fraction of Si

Self-diffusion coefficient of Al

*A. Falahati, International Journal of Materials Research 101 (2010) 1089–1096.

Phase Field Model

Vacancy diffusion coefficient Modified due to excess of vacancies *

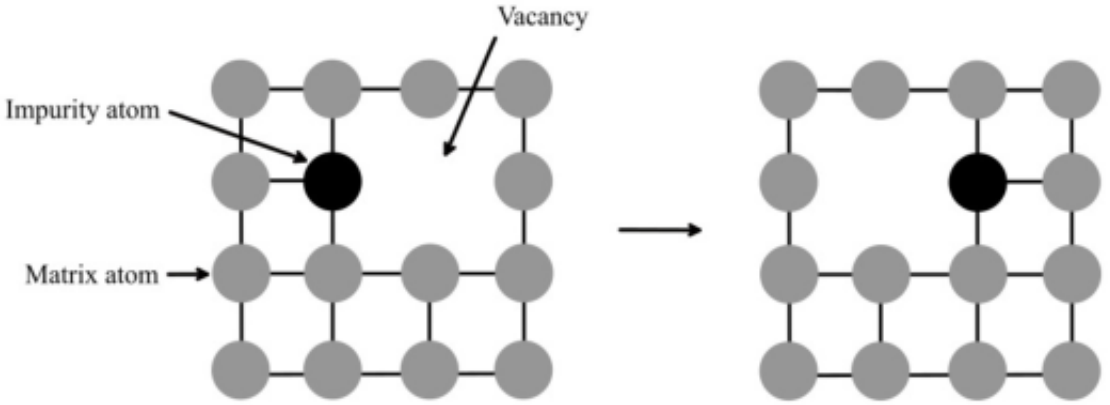
$$D_{Va} = \frac{X_{Va}}{X^e_{Va}}(X_{Si}{}^*D^{Al}_{Si} + (1 - X_{Si})^*D^{Al}_{Al})$$

Modified impurity diffusion coefficient of Si in Al

Original impurity diffusion coefficient of Si in Al

$$^*D^{Al}_{Si} = \left(\frac{X_{Va}}{X^e_{Va}}\right)\left(^*D_0{}^{Al}_{Si} exp\left(-\frac{Q_{^*D^{Al}_{Si}}}{RT}\right)\right)$$

Vacancy site fraction at equilibrium



*A. Falahati, International Journal of Materials Research 101 (2010) 1089–1096.

Phase Field Model

Governing equations

- Cahn-Hilliard for conserved field (molar fraction of Si in Al)

Al/Si Inter-diffusivity

Non homogeneous free energy

$$\frac{\partial X_{Si}}{\partial t} = \nabla . \left(\frac{\tilde{D}}{\partial^2 f_0 / \partial X_{Si}^2} \nabla \frac{\delta \mathcal{F}}{\delta X_{Si}} \right)$$

Homogeneous free energy

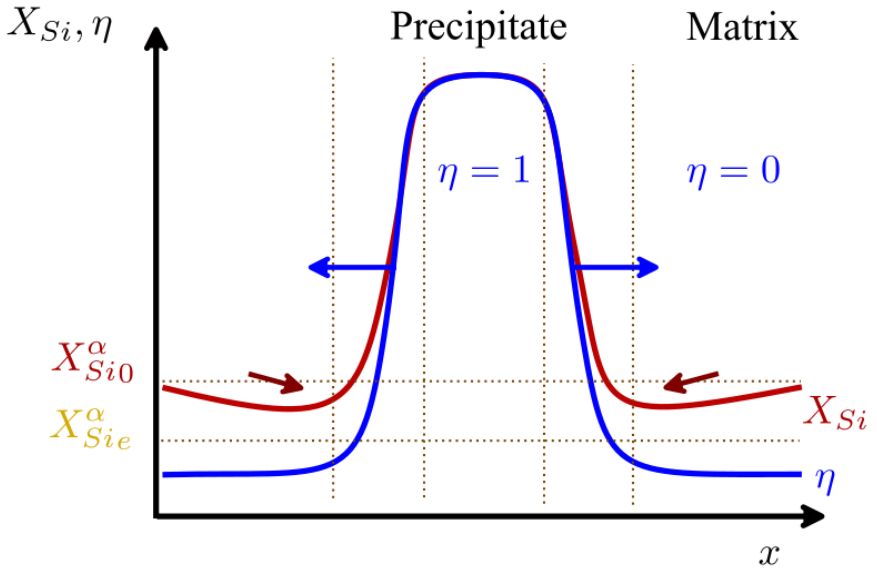
- Allen-Cahn for non-conserved field (phase)

Interface kinetic coefficient

$$\frac{\partial \eta}{\partial t} = -L \frac{\delta \mathcal{F}}{\delta \eta}$$

- Solved by Fourier spectral methods

Molar fraction of Si, Phase

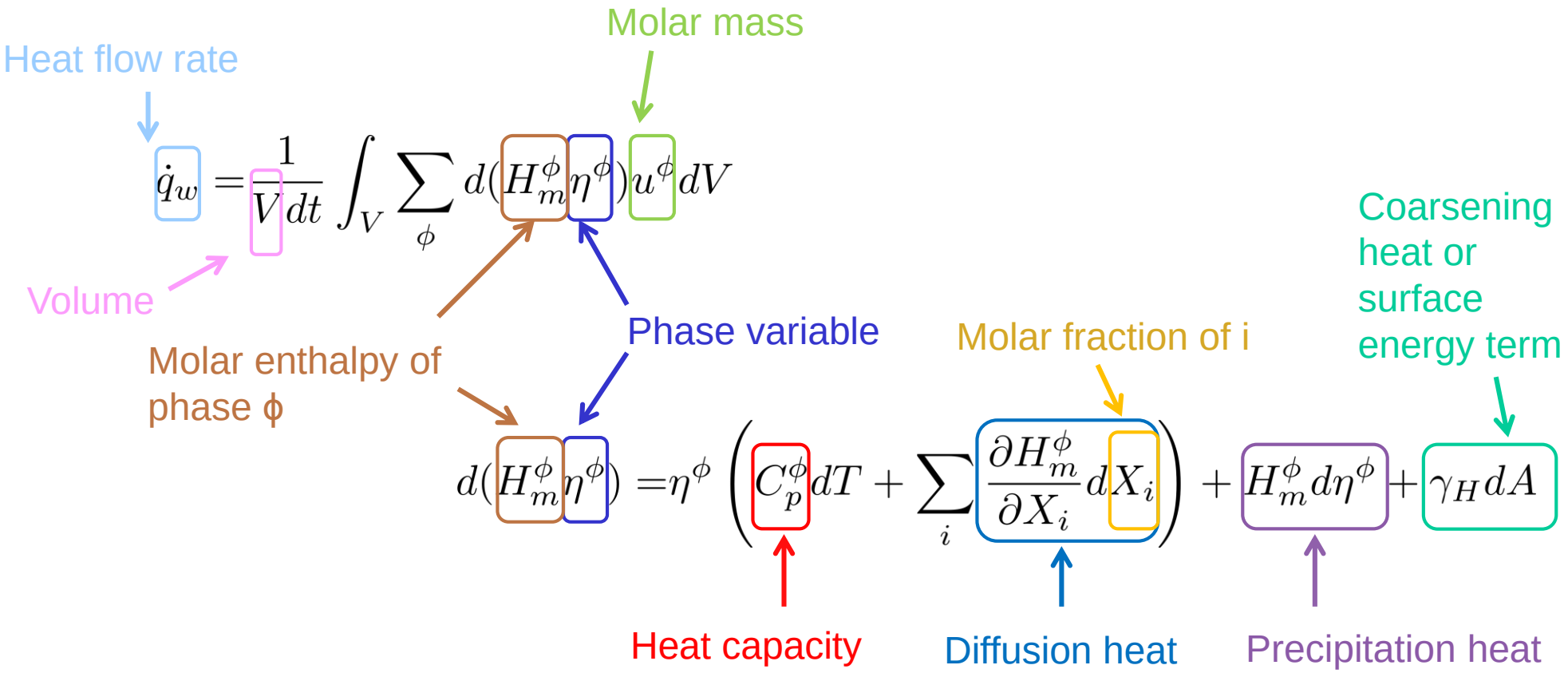


Evolution between initial out-of-balance system 0 and equilibrium one e

J. Zhu Physical Review E 60 (1999) 3564–3572.

1st validation: simulation of DSC curve

Heat flow rate absorbed / released by precipitate growth and coarsening



Model parameters

Model parameter	Symbol	Simplification	Tool / experiment	Reference
Free energy density	f^{α}, f^{d}	Parabola fitting	CALPHAD modeling	[ADR98]
Al/Si Inter-diffusivity	$\tilde{D}$	Al/Si Impurity diffusion coefficient		[Man+09]
Al Self-diffusivity	${}^{*}D^{Al}_{Al}$			[Man+09]
Interfacial mobility	M_{η}		DSC experiment	In-house experiment
Al/Si interface energy	γ		Back calculation from nucleation rate experiment	[RT58]
Initial conditions (phase fraction and molar fraction of Si)	η^{0}, X^{0}_{Si}		XRD + SEM analysis	In-house experiment
Molar volume	V_m		CALPHAD modeling	[Hal07]
Stiffness tensor	C_{ijkl}	Use Aluminum value for the whole system	CALPHAD modeling	[Su+15]
Equilibrium vacancy site fraction	X^{e}_{Va}			[Meh07]

[ADR98] I. Ansara, A.T. Dinsdale, M.H. Rand, COST 507 - Definition of Thermochemical and Thermophysical Properties to Provide a Database for the Development of New Light Alloys, 1998.
[Man+09] M. Mantina, Y. Wang, L.Q. Chen, Z.K. Liu, C. Wolverton, Acta Materialia 57 (2009) 4102–4108.
[Hal07] B. Hallstedt, Calphad 31 (2007) 292–302.
[RT58] H.. Rosenbaum, D. Turnbull, Acta Metallurgica 6 (1958) 653–659.
[Su+15] D. Su, Y.-L. He, J.-Q. Liu, X.-G. Lu, ICISMME, 2015.
[Meh07] H. Mehrer, Diffusion in Solids: Fundamentals, Methods, Materials, Diffusion-Controlled Processes, Springer Science & Business Media, 2007.

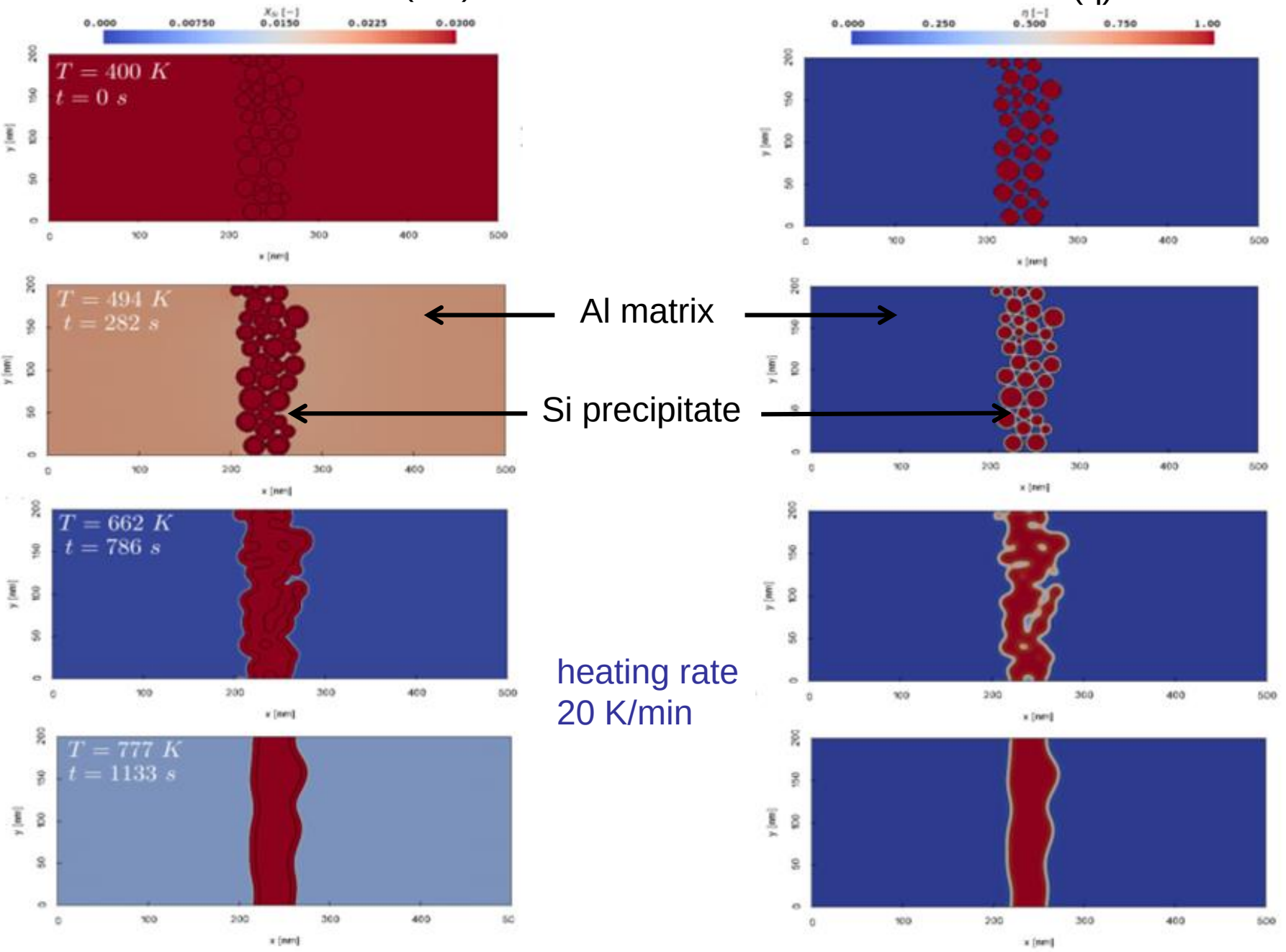
First simulation: sensitivity to L
Interface kinetic coefficient

- Domain size 200x500 nm, grid size 1 nm
- Maximum value of the interface kinetic coefficient L with the temperature **to ensure stability**
- L_{max} = a exp (bT) with a=3.63x10^{-18} and b=0.025
- Small value of L: microstructure evolution is interfaced-controlled with a low transformation kinetic.
- Large value of L: microstructure evolution is diffusion-controlled with a higher kinetic
  especially when the temperature is high.

2D simulation of a Si precipitate wall

Molar fraction of Si (Xsi)

Phase (η)



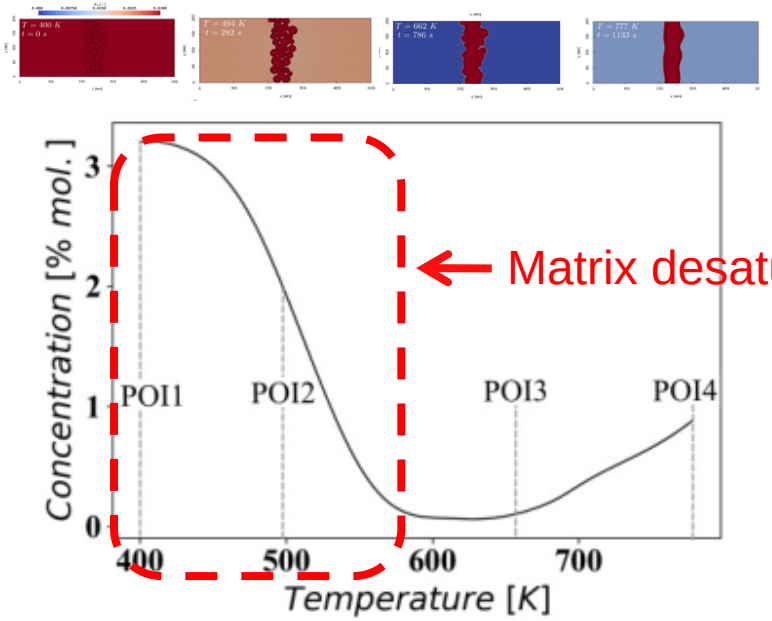
Al matrix

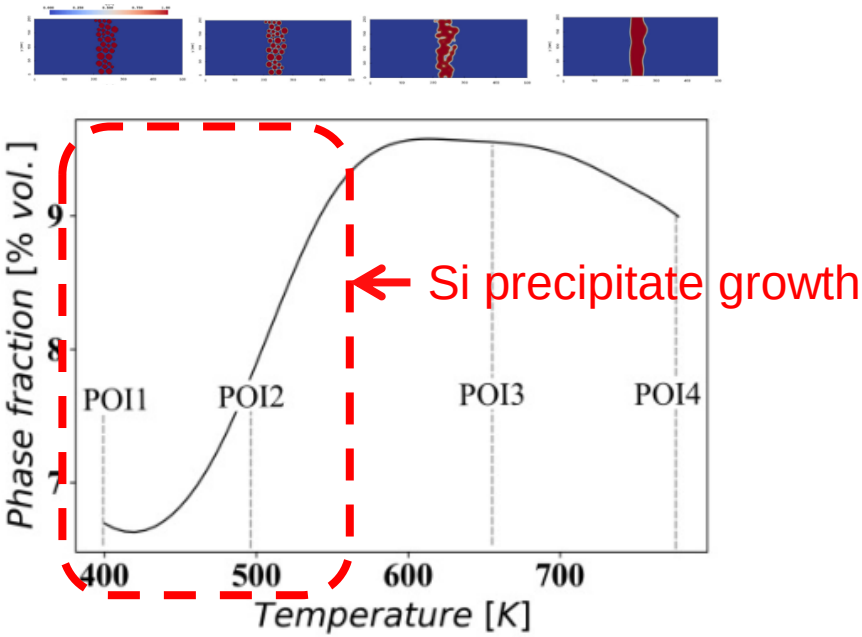
Si precipitate

heating rate
20 K/min

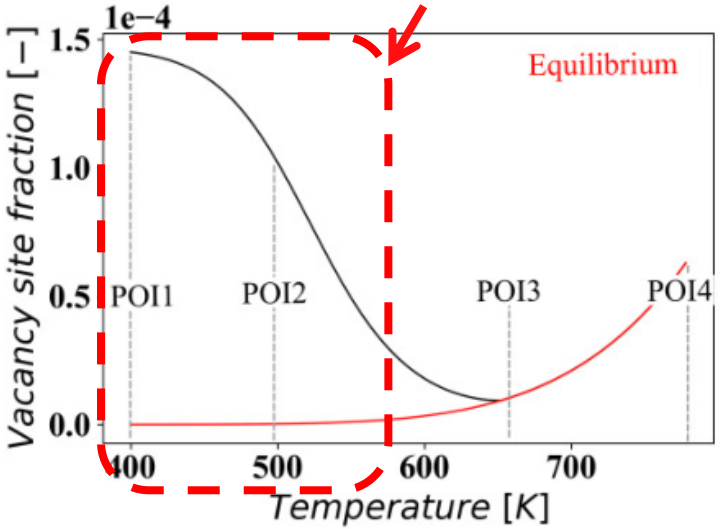
2D simulation of a Si precipitate wall

Evolution of average phase-field variables

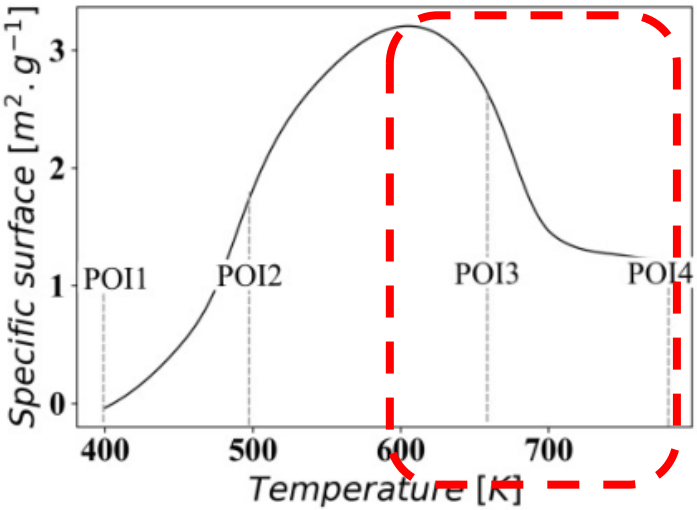




Solute diffusion enhanced by excess vacancies



Si precipitate coalescence / coarsening

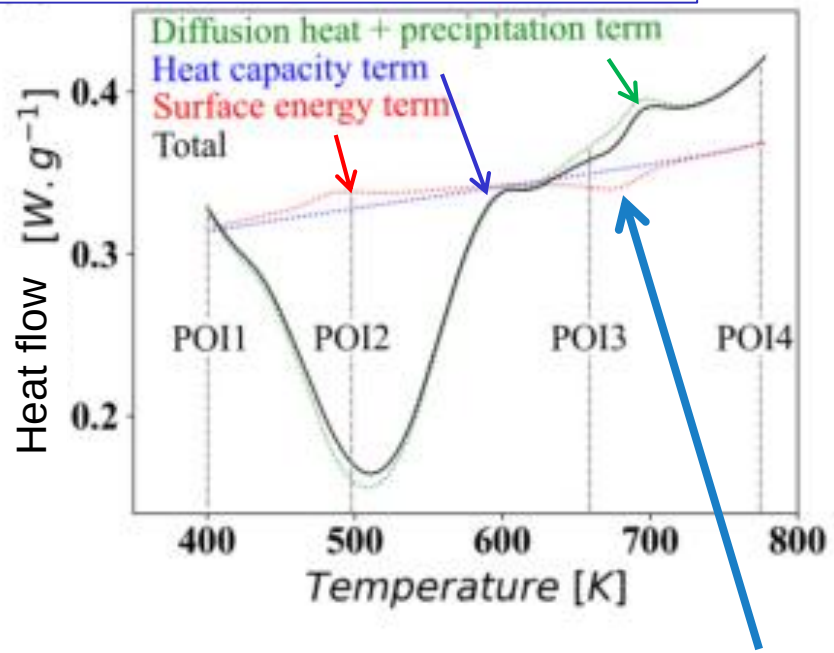


2D simulation of a Si precipitate wall

Differential Scanning Calorimetry (DSC) prediction vs experiment

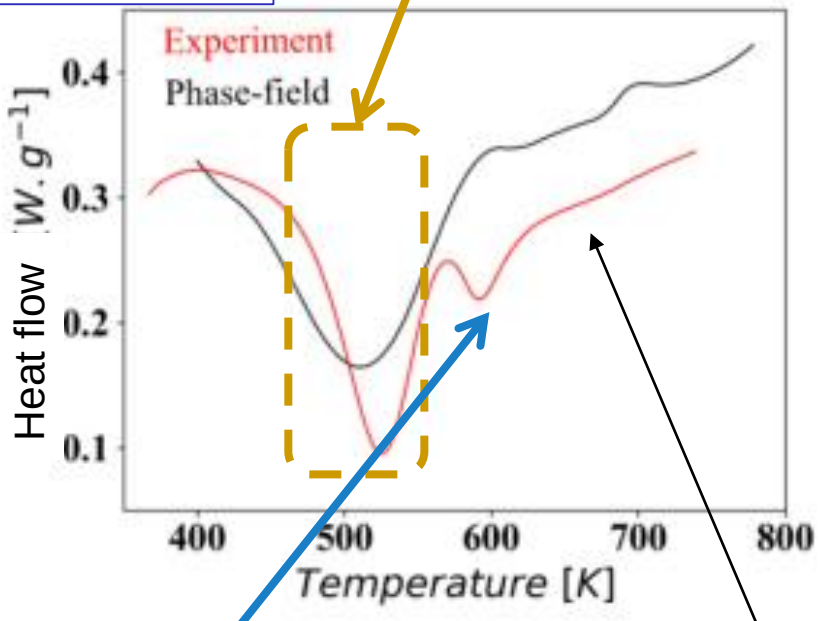
- Discrepancy with the baseline (heat capacity term) : heat peak area calibration ≠ heat flow rate calibration for DSC device

Phase Field prediction



Compared with exp.

First peak (desaturation of Al matrix with Si) well simulated

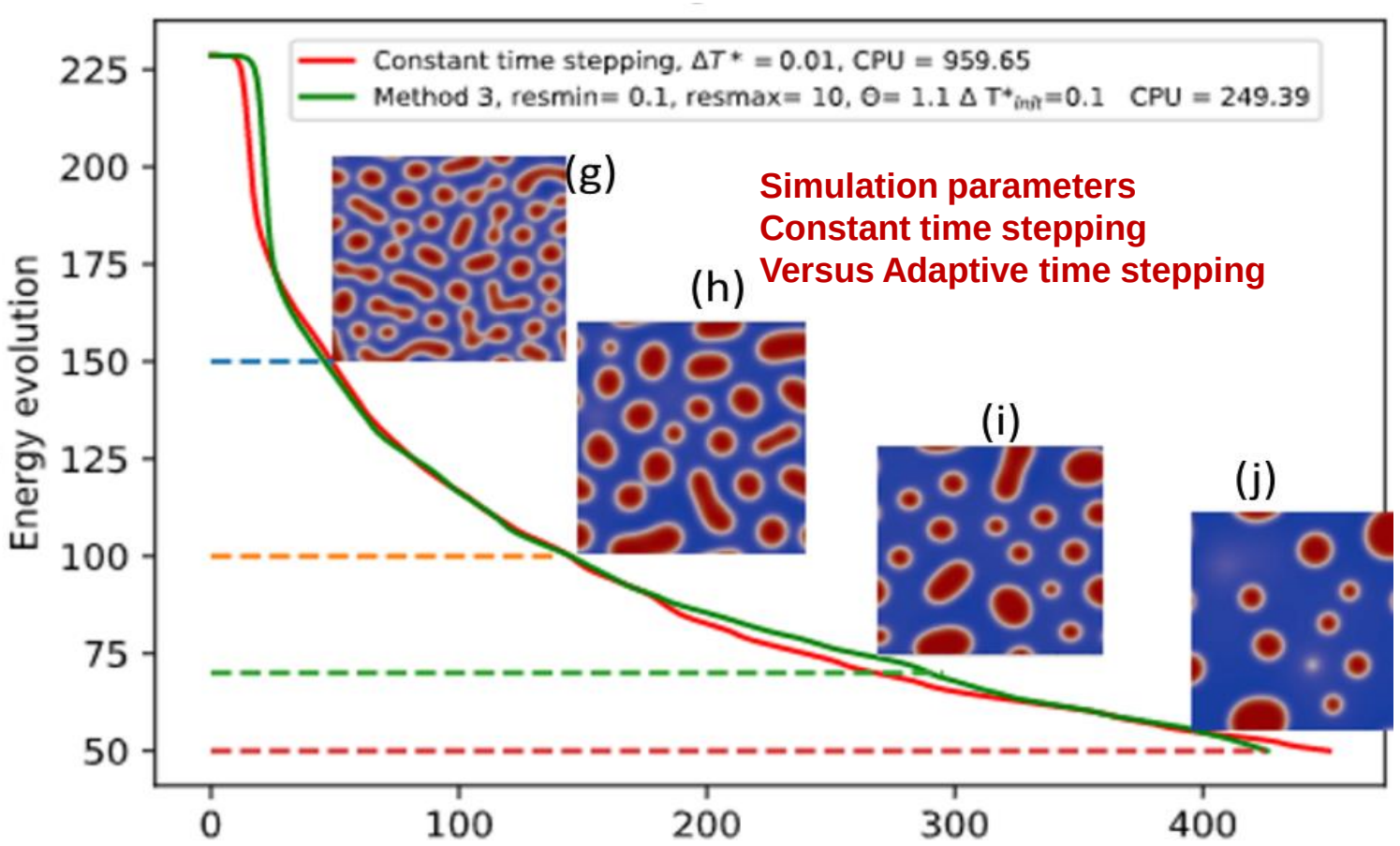


Second peak (Si precipitate coarsening) shifted to high temperature -> need to tune model parameters

Considering base line, experiment calibration not perfect so red line will ↑

Phase-Field simulations Adaptative time stepping

- To ensure and control the stability of the simulation.
- To speed up the simulation.

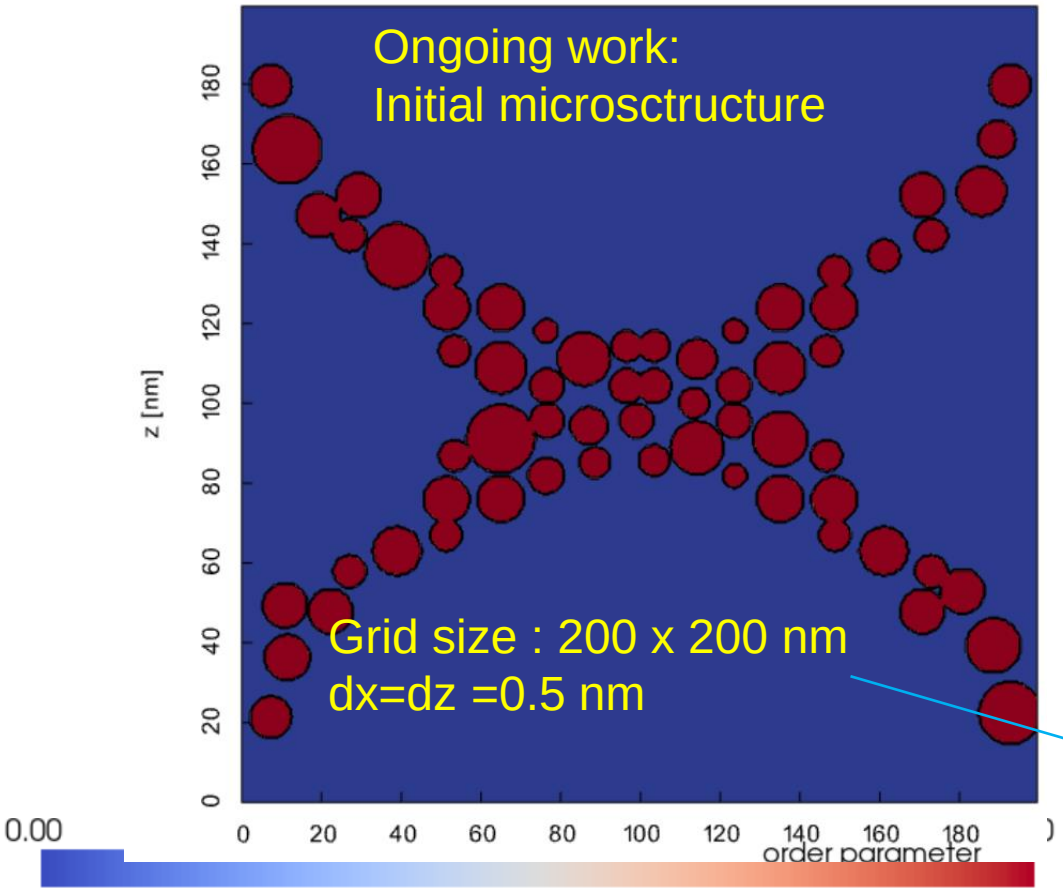


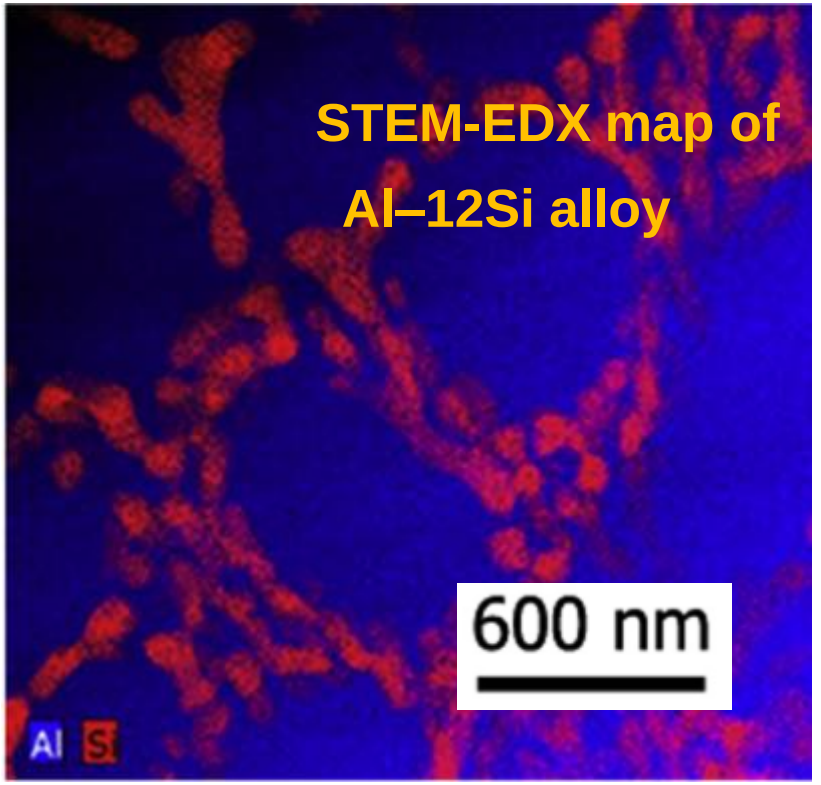
Solving
Cahn Hilliard
Conservative
Equation

S. Fetni , J. Delahaye , L. Duchêne , A. Mertens, A.M. Habraken ; Adaptive time stepping approach for Phase-Field modeling of phase separation and precipitates coarsening in additive manufacturing alloys - COMPLAS 2021

2D simulation DSC1st run (heating rate 20 K/min) evolution of Si precipitate wall

-no limitation on kinetic coefficient L
→ confirmation interface controlled microstructure evolution
-time step variable (3X smaller if evolutions occur)





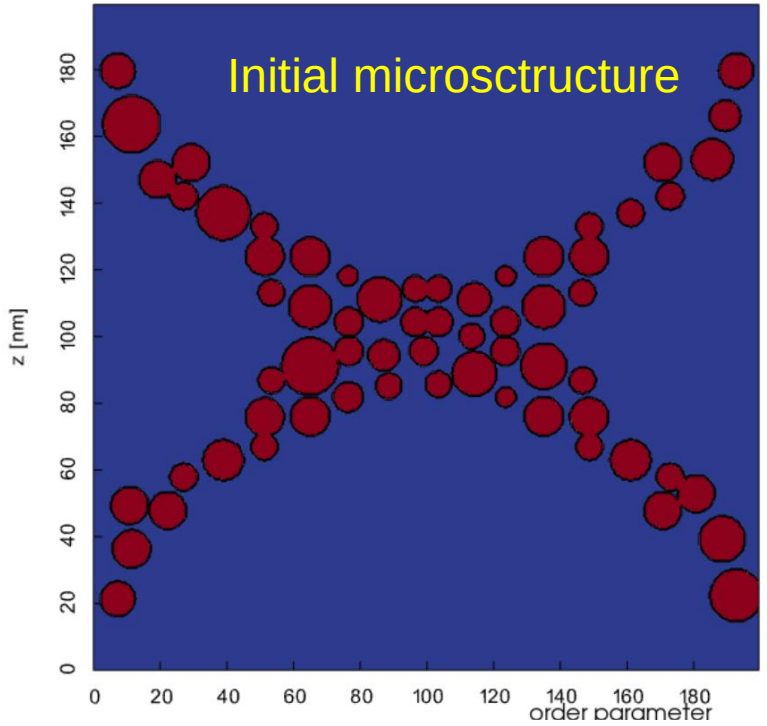
X.P. Li et al., Acta Mater. **95** (2015), 74-82

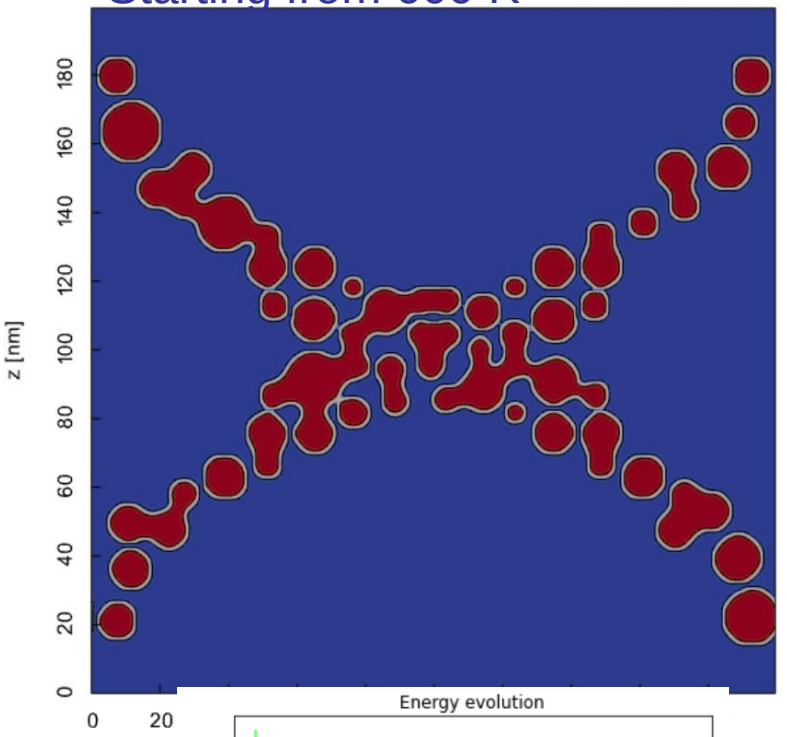
To increase to 500 x 500 nm (periodic cell)

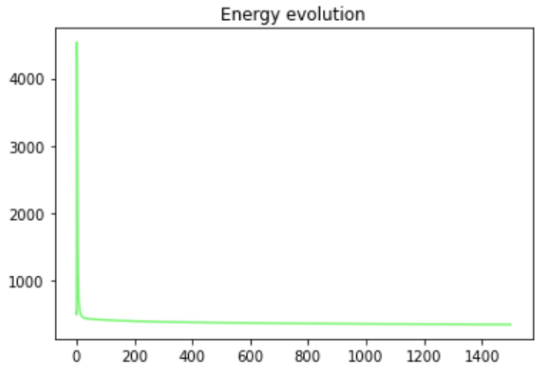
2D simulation DSC1st run (heating rate 20 K/min) evolution of Si precipitate wall

Simulation of a Representative wall of AlSi10Mg cell

Anisothermal heating (20 K/min)
Starting from 600 K

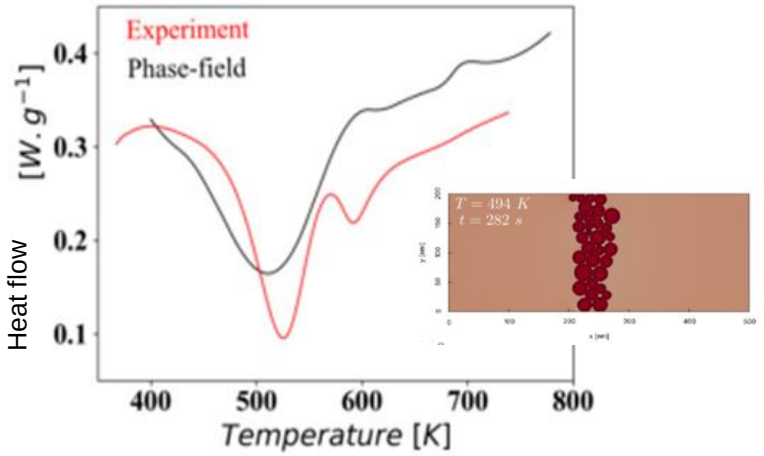


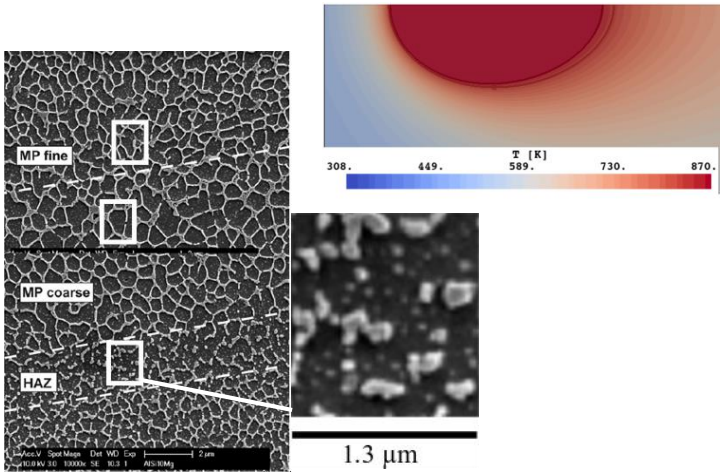




Conclusions - Phase Field

- KKS model + effect of excess quenched-in vacancies on Si solute diffusion
- Si precipitate growth and coarsening kinetics is predicted
- 1st simu agreement between predicted / experimental DSC curve Peak 1
- 2nd simu to predict Peak 2 related to Si precipitate coarsening





Transformation kinetics of bulk AlSi10Mg under a heating rate of 20 K/min (DSC conditions)

GOAL

Transformation kinetics heating/cooling rate of 10^6 K/s (L-PBF conditions)

Conclusions - Phase Field - FE

Iterative loop ?

Microstructure evolution

New thermo-physical properties

New Temperature history

FE model

Temperature history

Phase Field

Microstructure evolution

Objective = microstructure prediction of As build L-PBF & As Built + heat treatments

Final Microstructure → Final Properties