

In-situ studies of phase transformations during welding using synchrotron radiation

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Outline

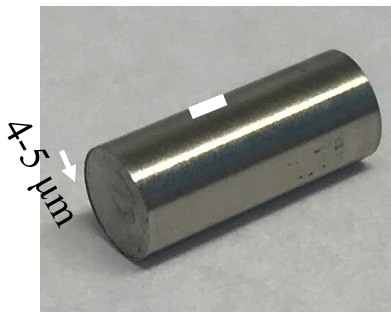
- Why synchrotron?
- Set up of the experiment
- Experimental data
- Results
 - Phase transition
 - Precipitate formation
 - Nirides
 - Sigma phase



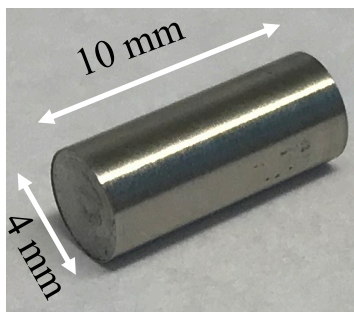
Advantage of synchrotron radiation

- **Non-destructive:** no particular sample prep. required
- **High energy:** Bulk penetration

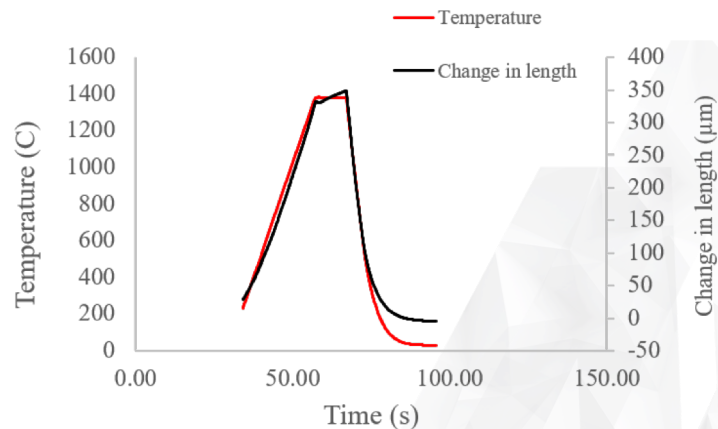
In-lab XRD



Synchrotron



- **High flux, high energy fast acquisition time:** access kinematic



Example: 10 XRD every second i.e. 150 C/sec we can have 15 C resolution

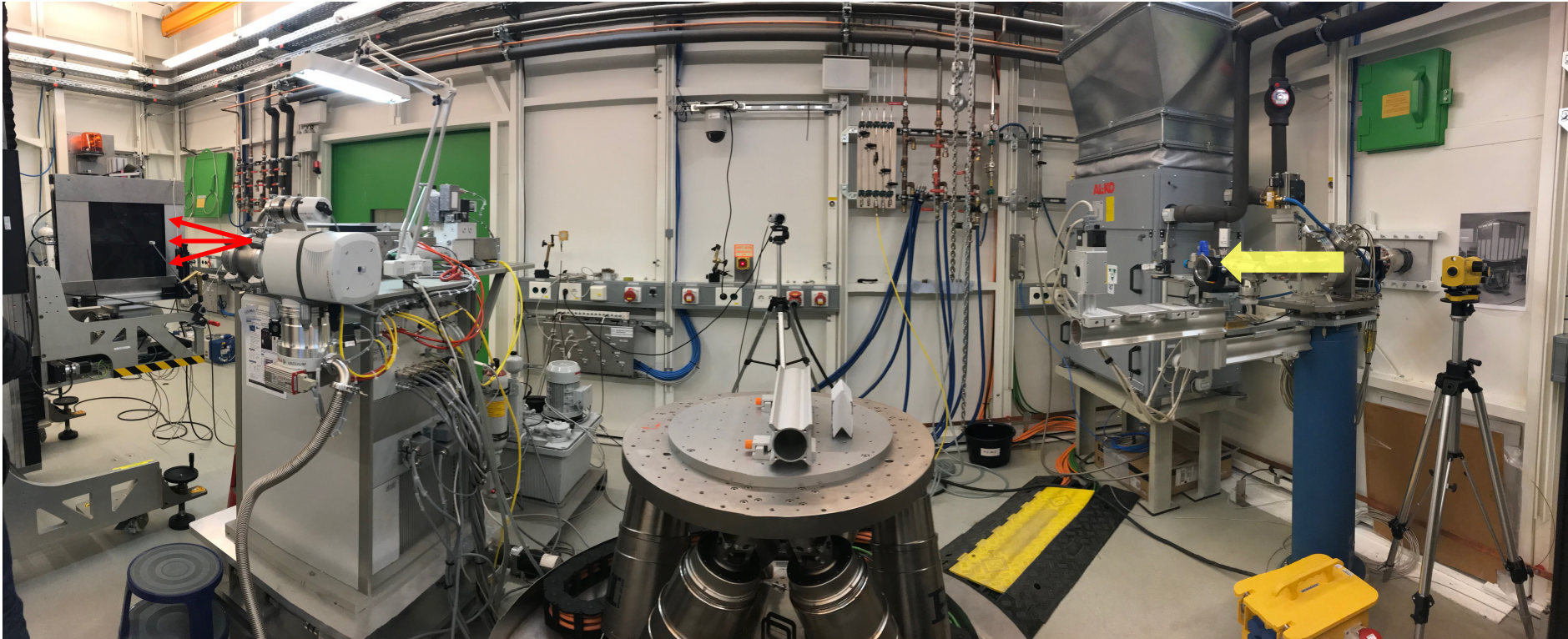
Experiment setup

SWERIM

Detector

Sample

Incoming beam



DESY, PETRA III, P07

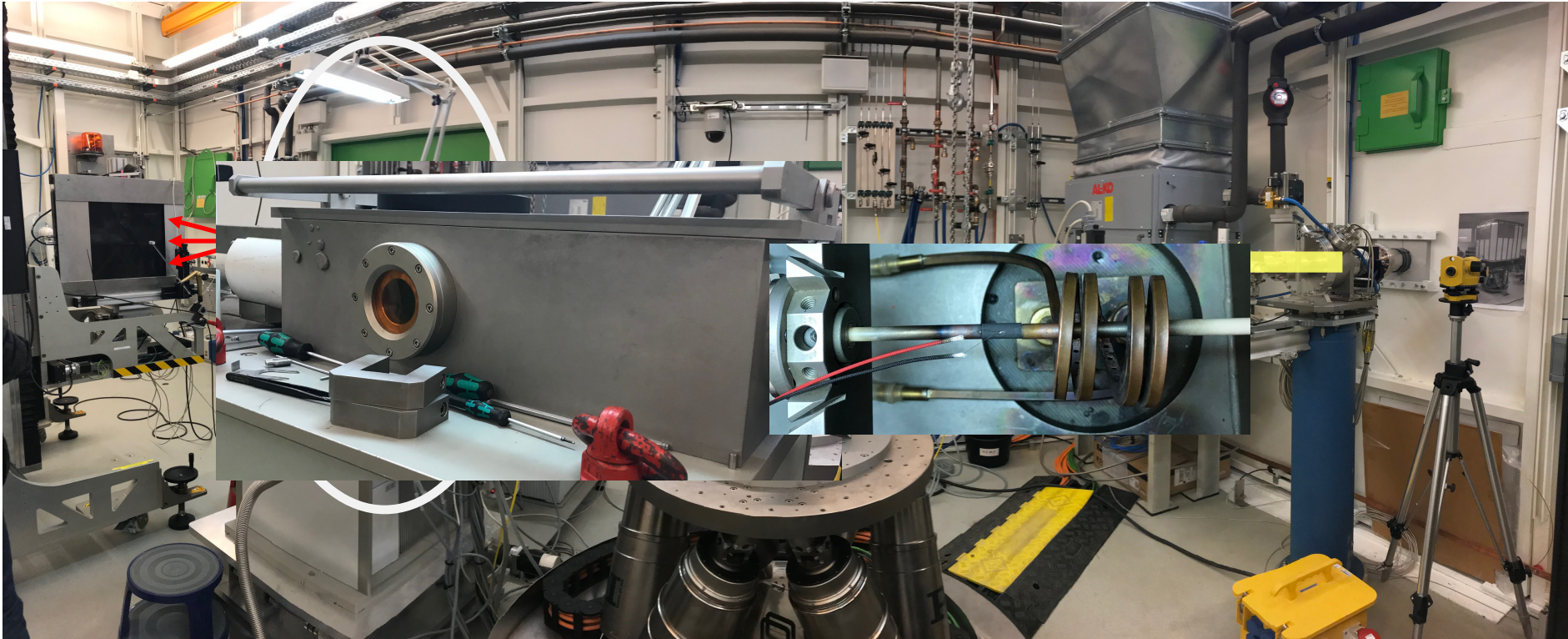
Experiment setup

SWERIM

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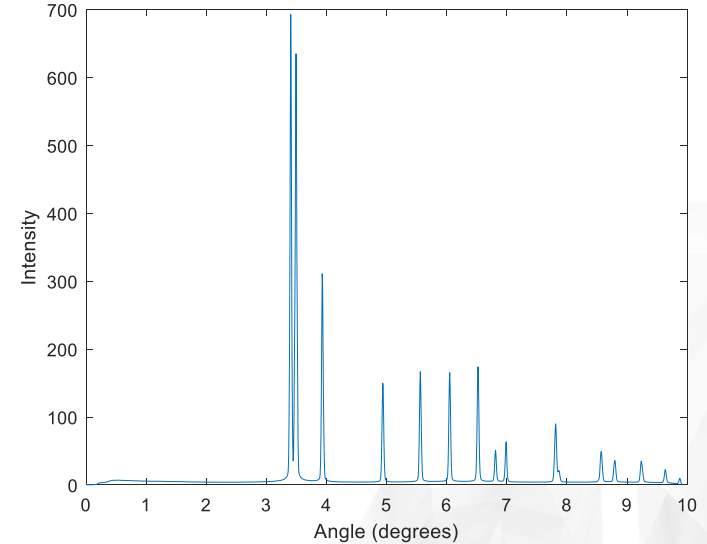
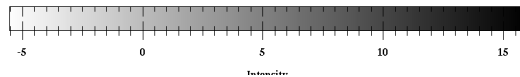
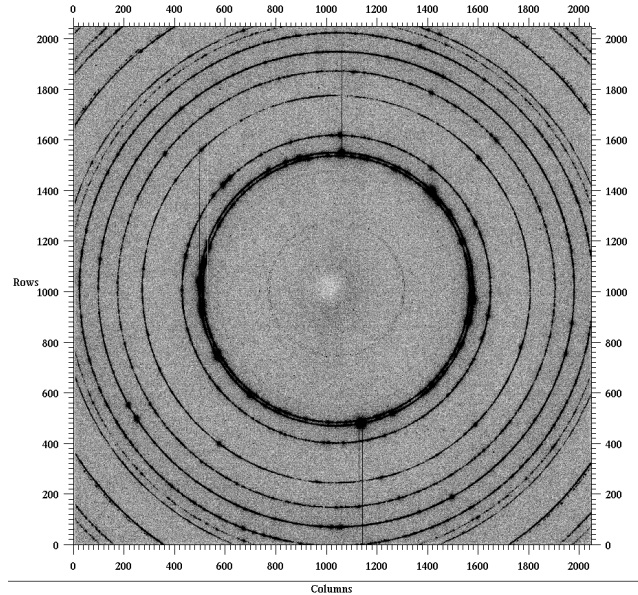
Incoming beam



DESY, PETRA III, P07

Raw data

Detector

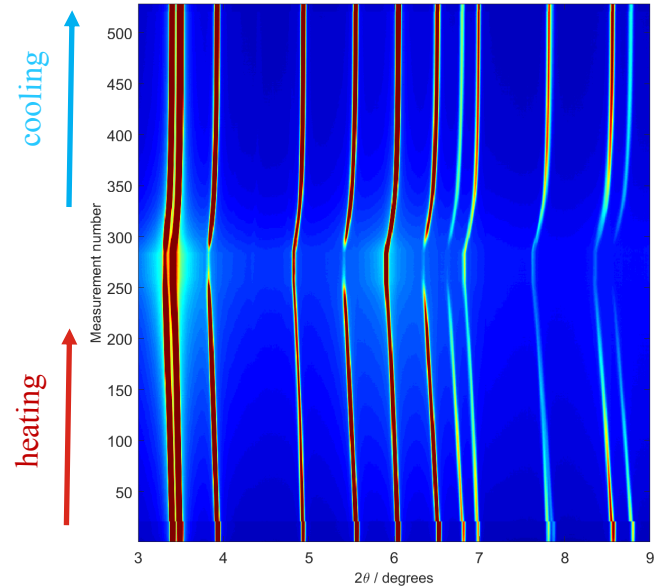
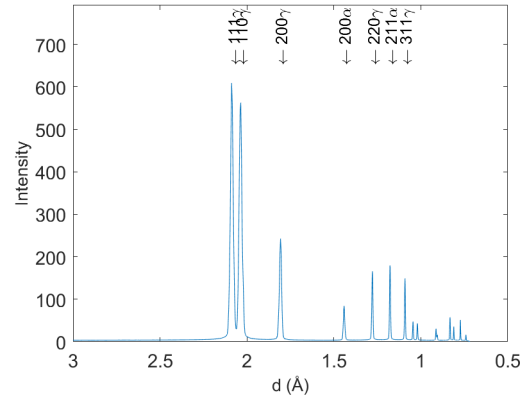
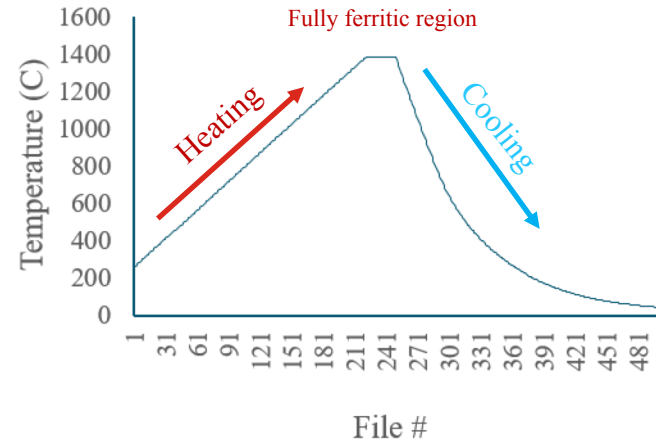


Peak position: lattice parameters

Peak width: strain/stress within the lattice

Intensity: phase fraction

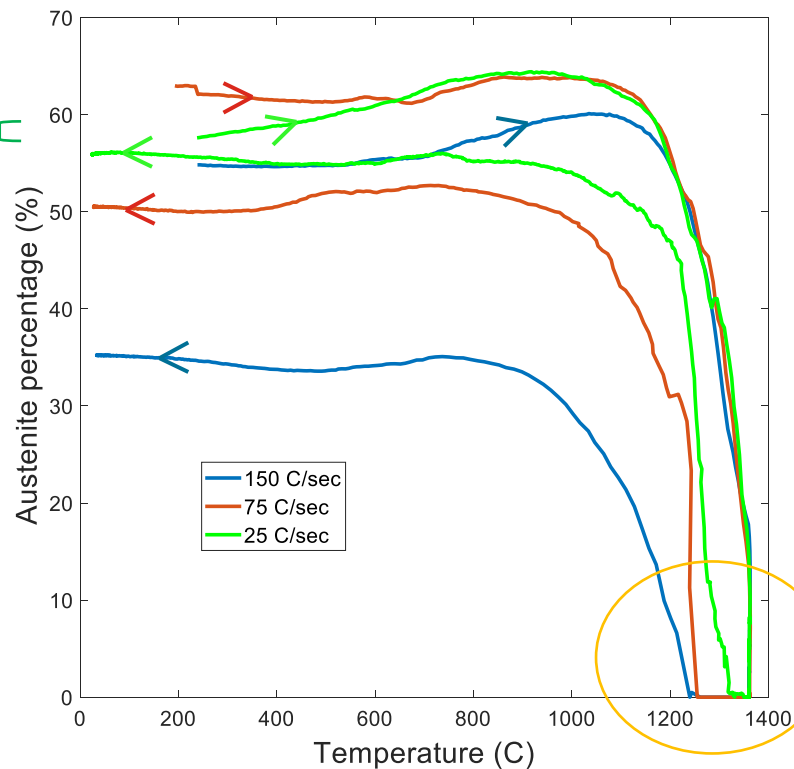
Diffraction data example



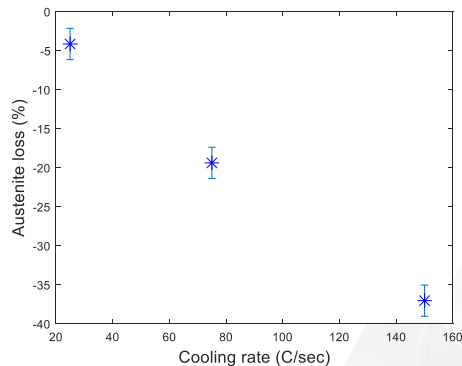
Such a cycle: ~ 630 one dimensional XRD

Phase fractions in-situ

10 second holding



Higher cooling rate: less austenite formation

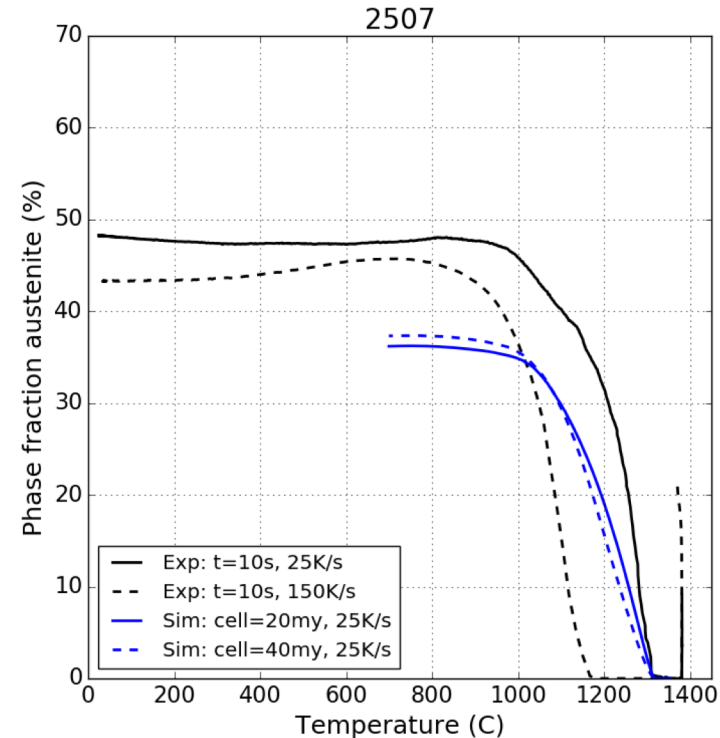


Fully ferritic region can be readily distinguished, and austenite formation temperature can be probed: accurate phase diagram and kinematic behavior

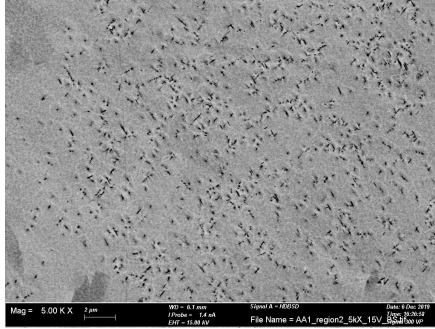
Phase evolution: simulation vs experiment

Austenite reformation during cooling:

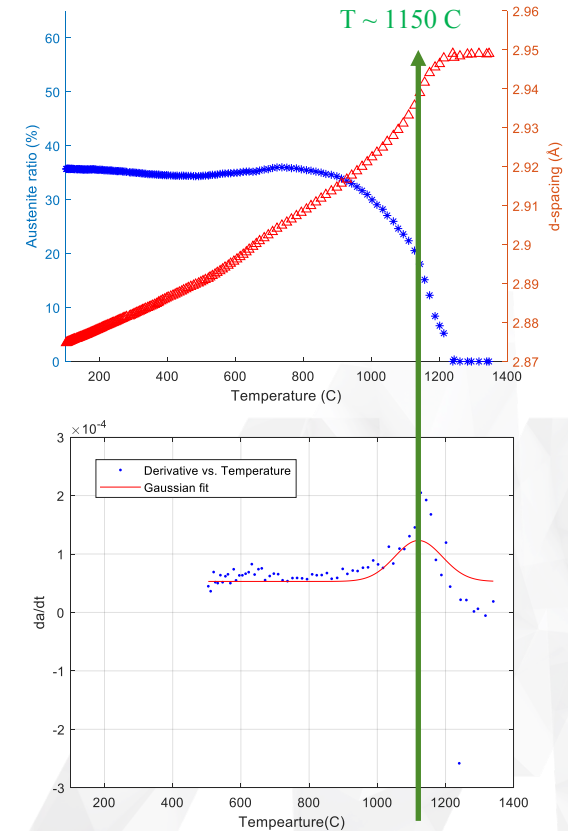
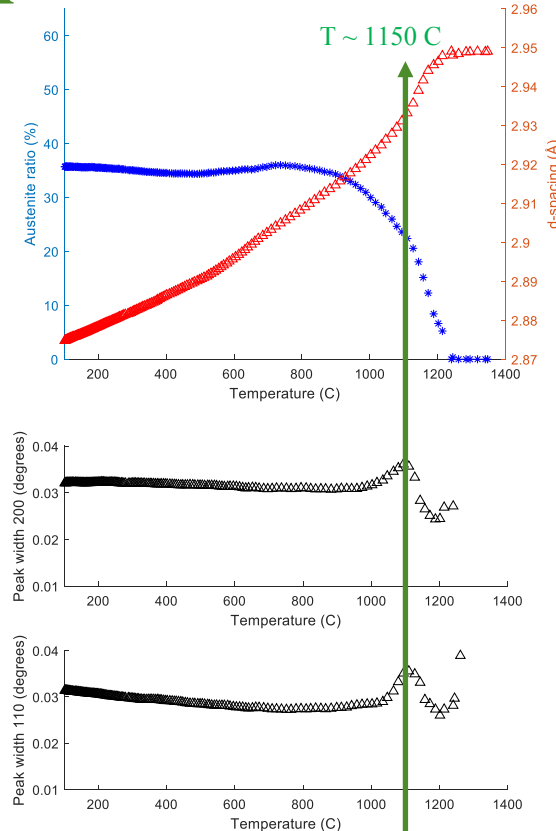
- ✓ Good agreement with austenite transformation T
- Simulation shows slower austenite formation compared to the experiment, to modify:
 - modeling is done in 1D and simulates planar growth of sigma
 - Initial microstructure is important
 - Stronger driving forces?



Nitride formation: low-quantity no detectable **SWERIM** diffraction peak



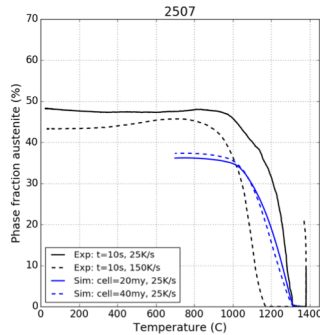
- Derivative of lattice parameter during the cooling shows a maximum at a certain point during cooling
- At this point the strain within ferrite matrix increases: induced by precipitate formation?



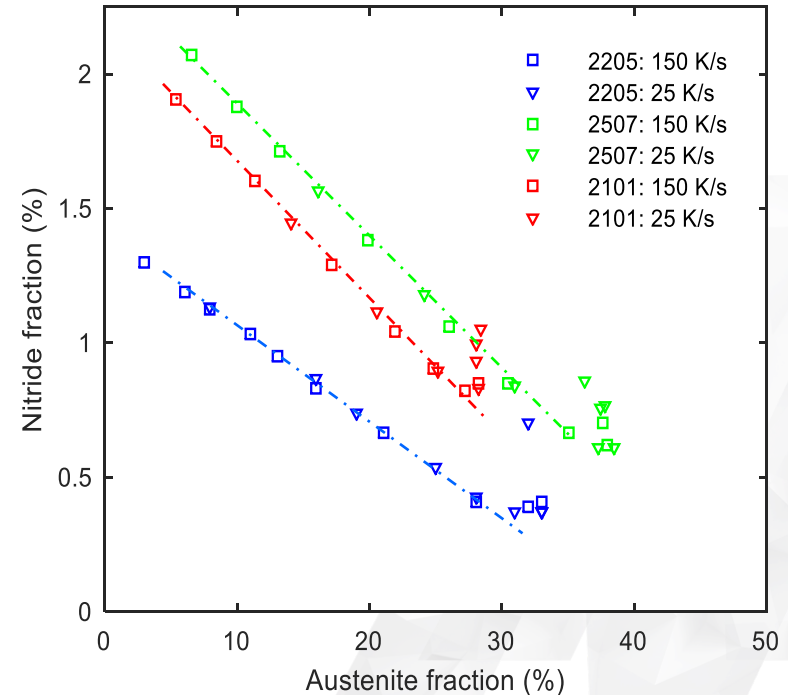
Possible signs of nitride formation (?)

Nitride quantification: relating experimental results with **SWERIM**

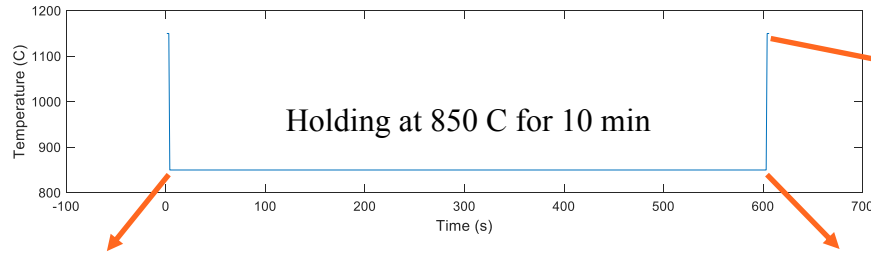
Fine tune the important simulation parameters to match the kinetics observed in experiment: i. e. better estimate of austenite quantity



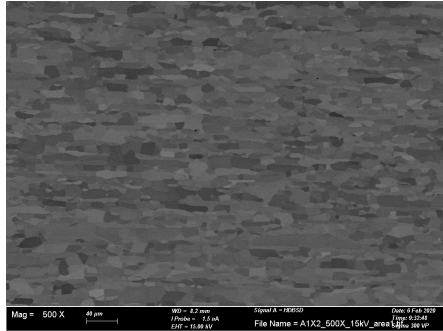
Correlating the austenite fraction with that of nitrides during the treatment



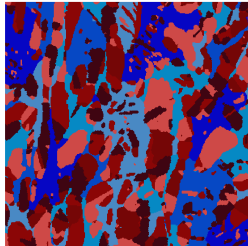
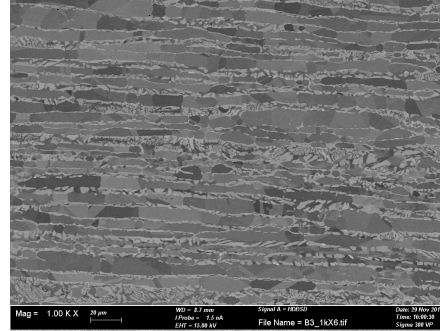
Intermetallic sigma phase



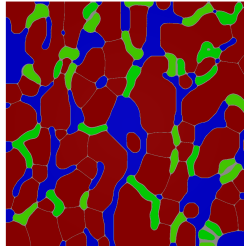
Heating to 1150 C to homogenize the sample



SEM

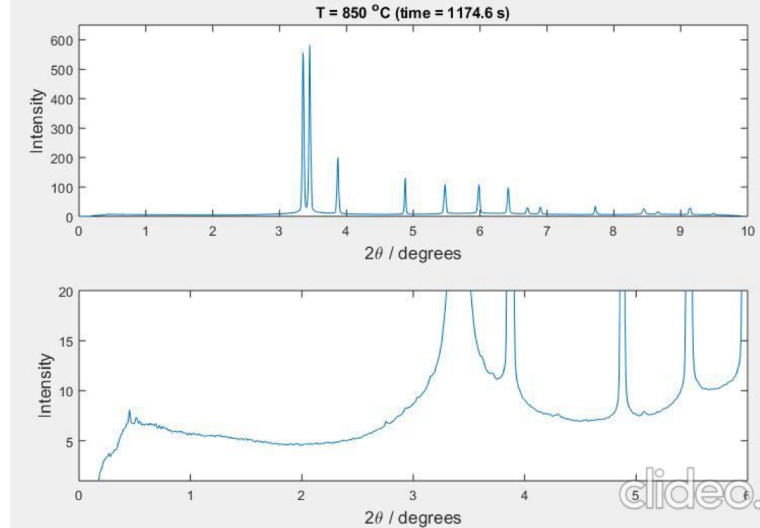


Initial structure (EBSD)



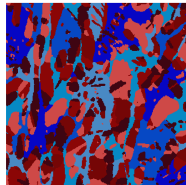
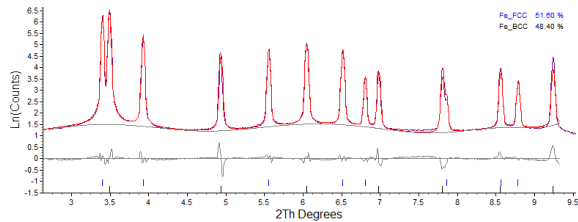
850°C for 1 hour

Fast-forward movie: 10 minutes holding at 850 C

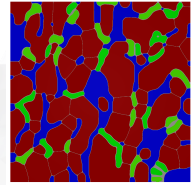
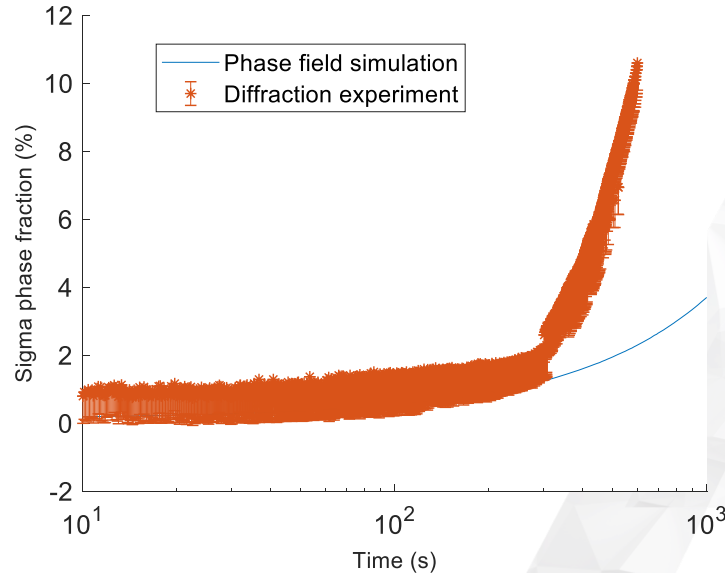
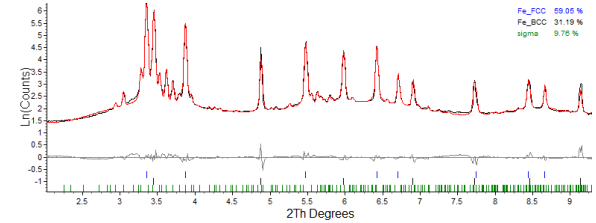


Diffraction experiment vs. phase field simulation

- Initial microstructure
- Size, area and austenite spacing
- History of the sample
- Fine tuning simulation parameters to allow faster growth



Initial structure



Held at 850 C

Summary

Use of synchrotron XRD

- ✓ Great tool for phase transformation studies in-situ
- ✓ Quantitative information for intermetallic phases
- ✓ Fast and accurate method to access kinetics and modify the simulation tools

Challenges:

- × Quantifying low fraction of precipitates like nitride → SAXS may be better
- × Understanding the data requires competence and can be time consuming

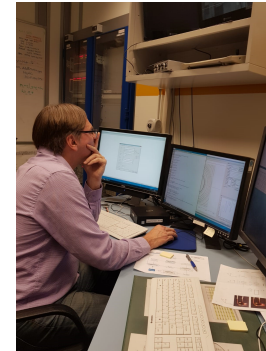
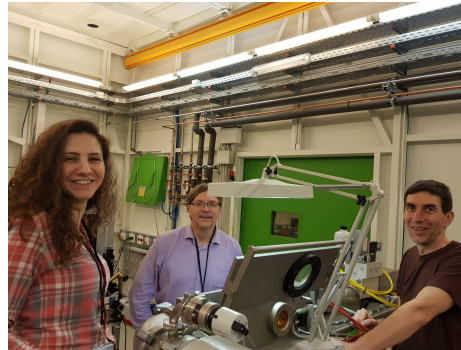
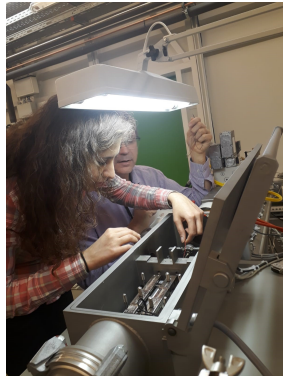
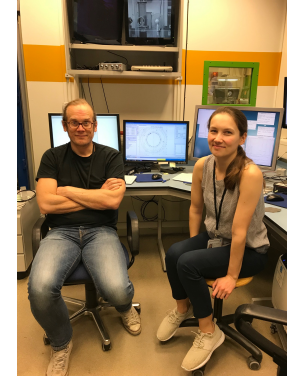
Thank you for your attention

SWERIM

and special thank to:

Vinnova funding

Jan Y. Jonson
Nataliya Limbach Malya
Fredrik Lindberg
Andreas Stark
David Lindell
Niklas Petersson
Amer Malik
Staffan Hertzman
Tuerdi Maimaitiyili
Lars

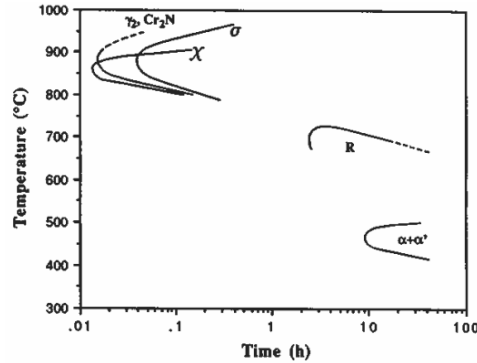




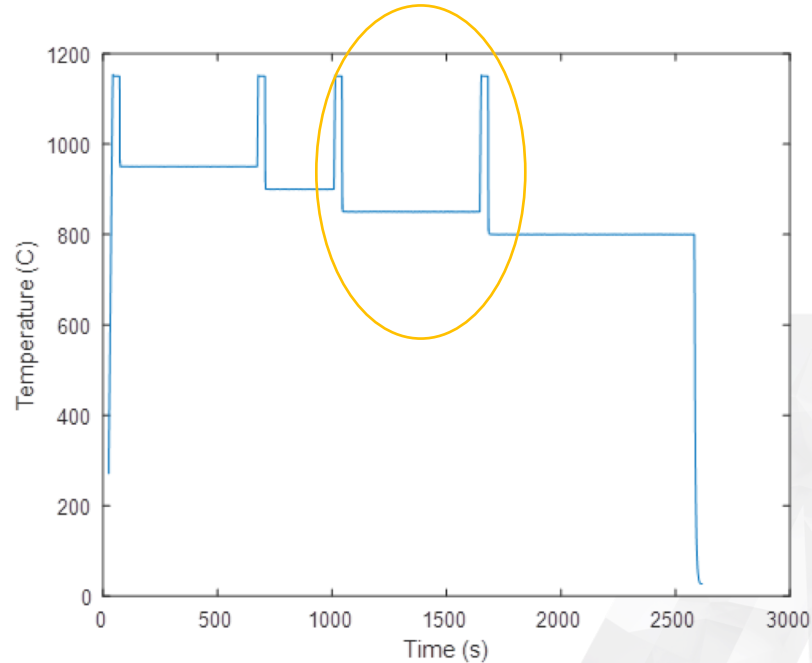
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Extra measurements

Sigma phase formation was seen in-situ



Nilsson, Materials Science and Technology 8, 685-700 (1992)

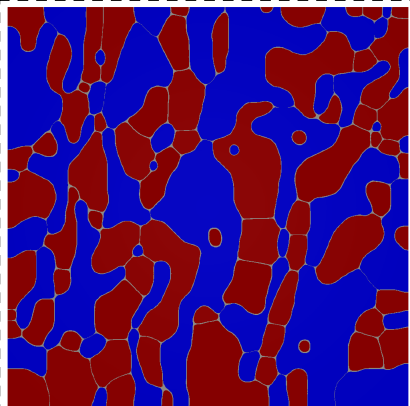


Analysis in progress

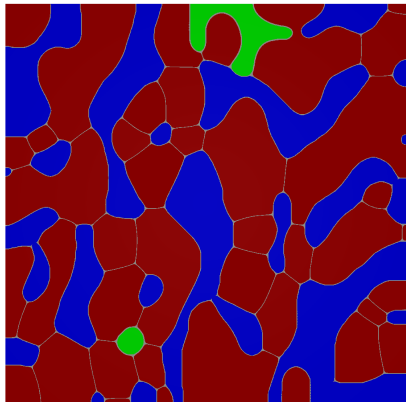
Results

■ Ferrite
■ Austenite
■ Sigma

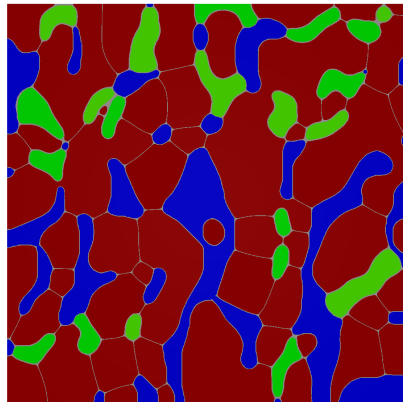
SWERIM



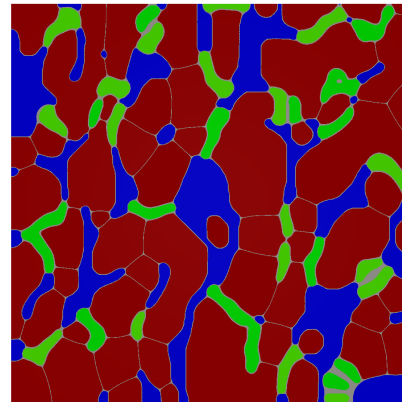
Initial microstructure



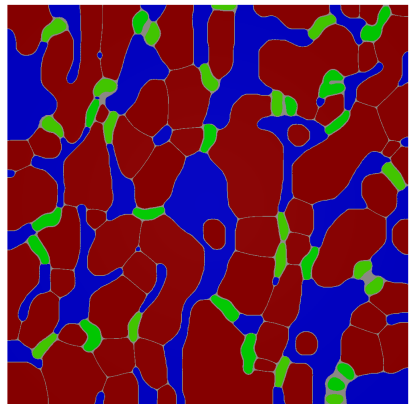
950°C for 2 hours



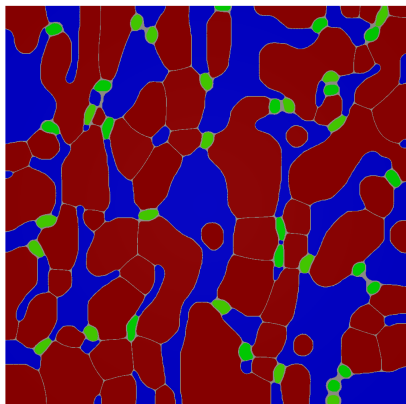
900°C for 1 hour



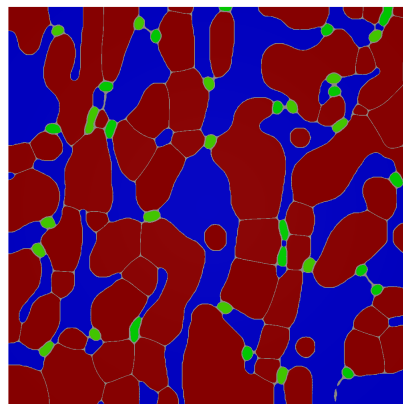
850°C for 1 hour



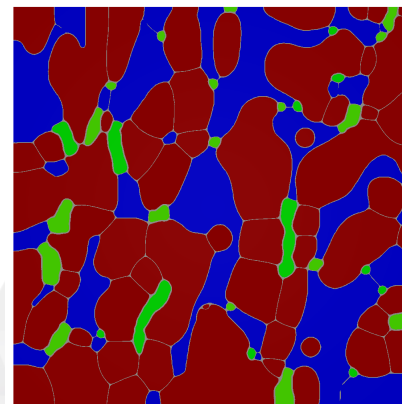
800°C for 1 hour



750°C for 1 hour



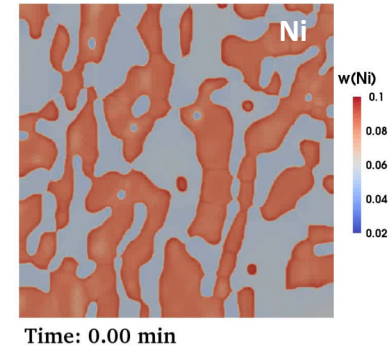
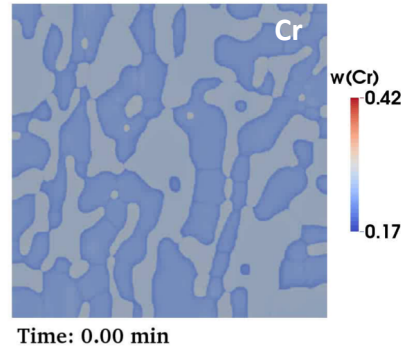
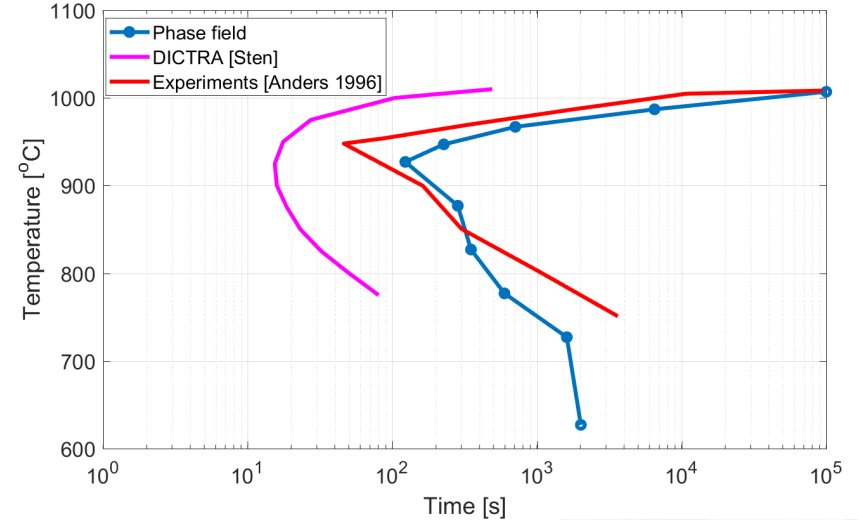
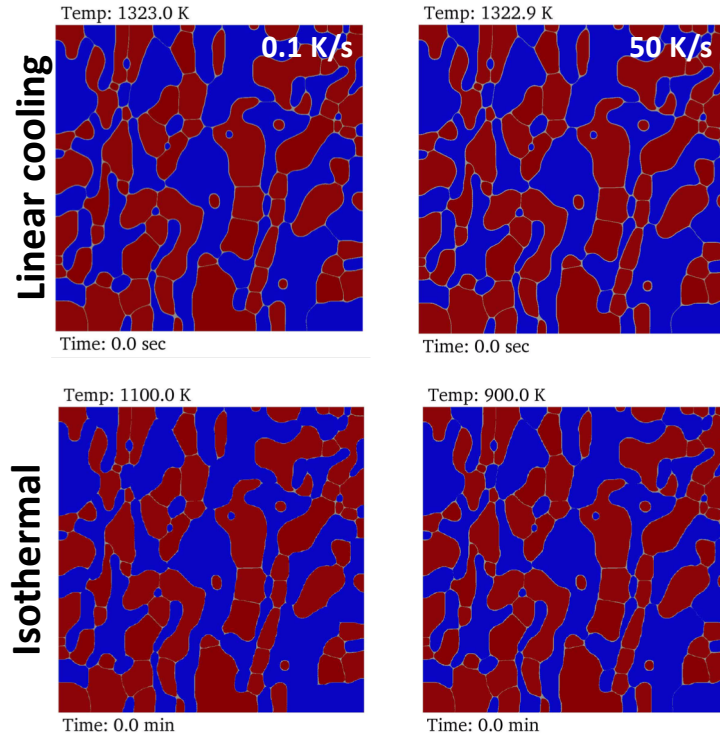
700°C for 2 hours



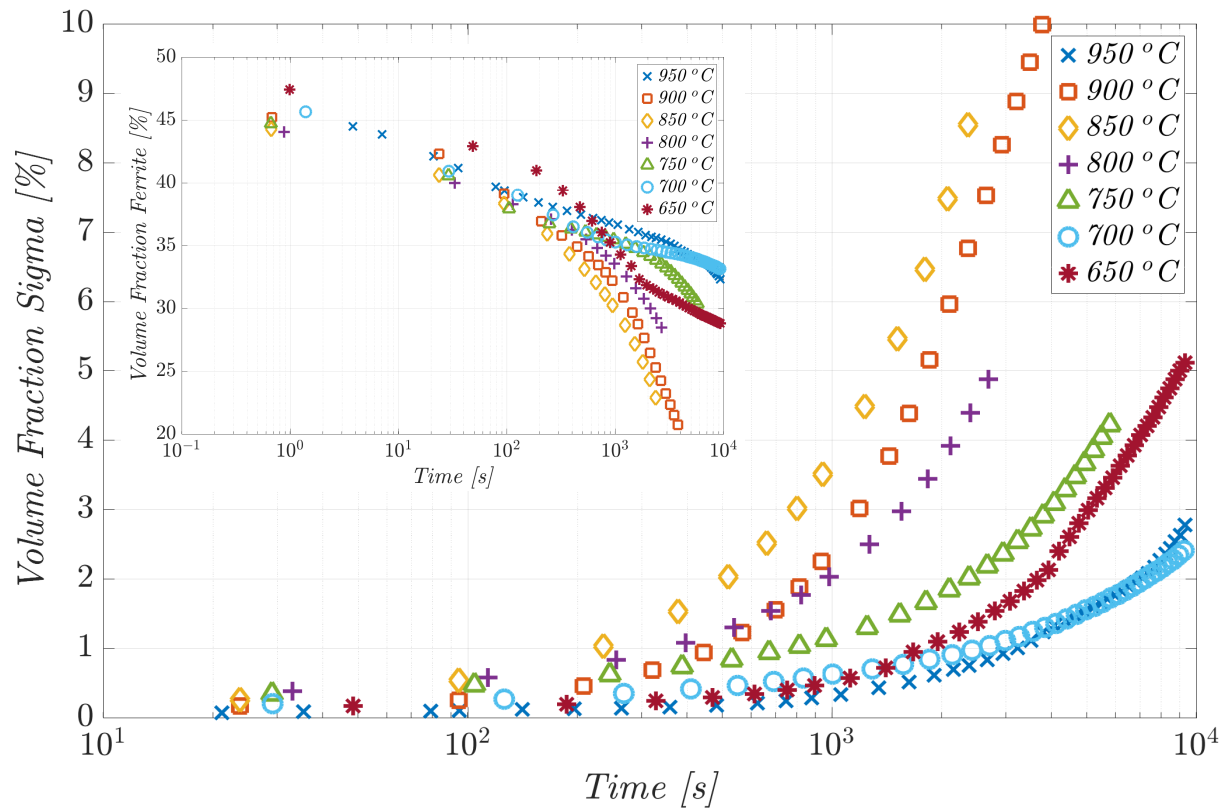
650°C for 2 hours

Example # 1

Nucleation and growth of sigma phase in SAF2507 super-duplex stainless steel alloy



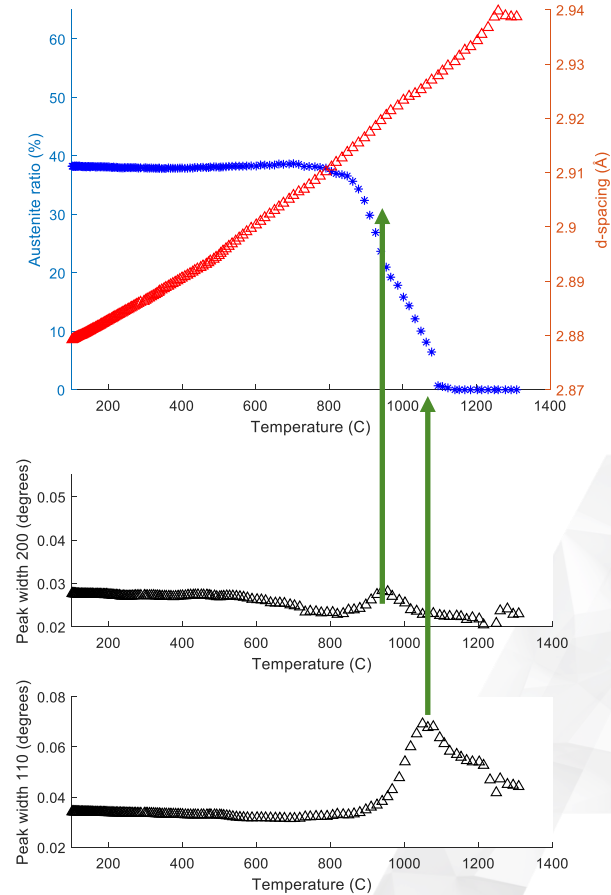
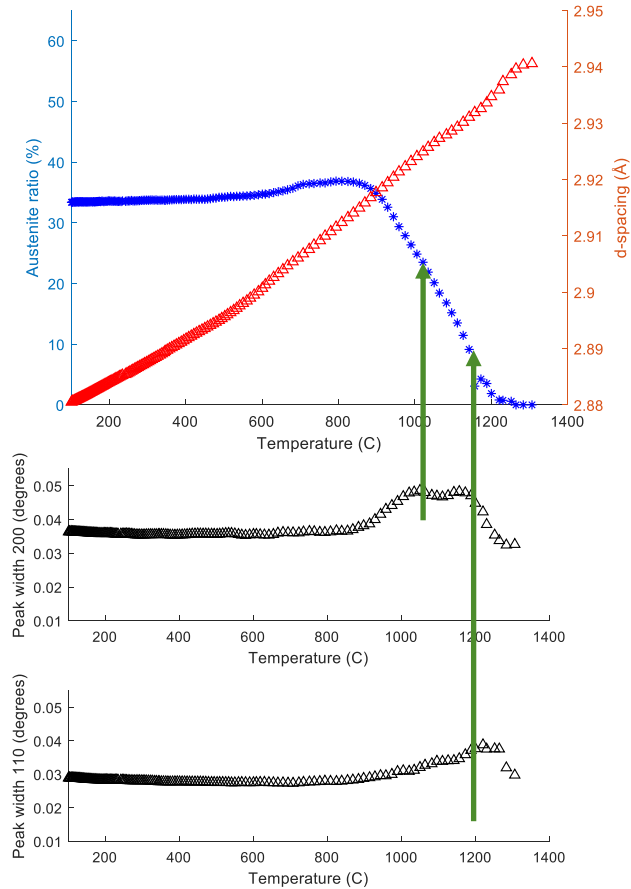
Phase field simulation



Indirect observation of nitride formation: 2205

3 sec at 1360 C and 150 C/sec cooling

10 sec at 1360 C and 150 C/sec cooling



2507 10 sec holding and 150
C/sec cooling

Derivative of 200 peak

