PhaNi-Neutron – Final Meeting

Phase and strain monitoring during direct metal deposition of Ni-superalloy by in-situ neutron and synchrotron diffraction (PhaNi-Neutron)

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Key Project Objectives

1. **Phase Precipitation Analysis:**
   Investigating phase kinetics during DED to better understand the relationships with process parameters, temperature distribution and microstructure.

2. **Strain Build-up Monitoring:**
   Utilizing neutron diffraction to characterize dynamic stress and strain evolution during the DED process.

3. **Traditional Laboratory Characterization:**
   Correlating microstructural details with in-situ and ex-situ measurements to enhance understanding of material properties.

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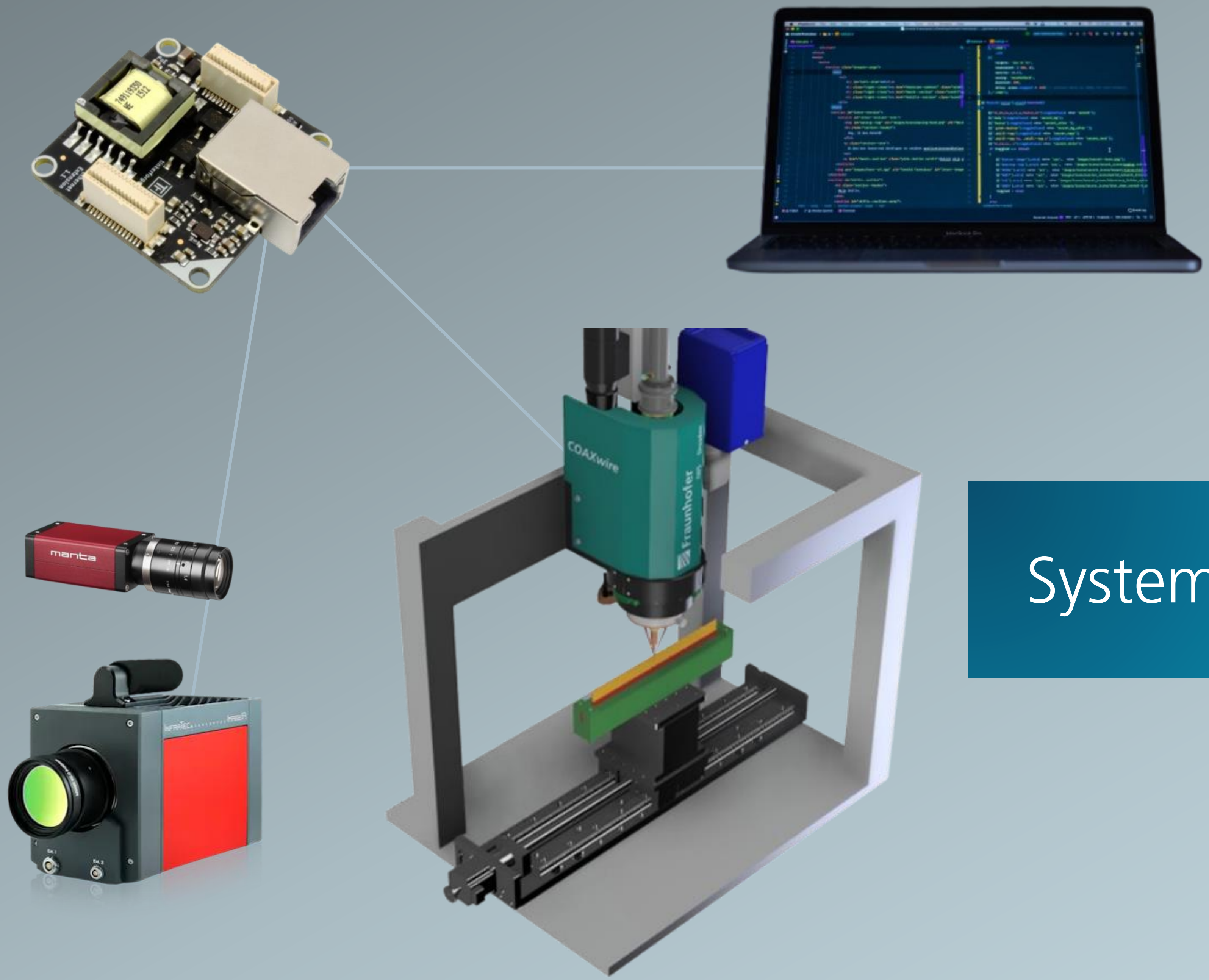
Main Tasks:

1. **System Interface Planning:**
   Development of improved system setups and interfaces for synchronized data acquisition, enhanced temperature monitoring and process repeatability.
2. **Material Characterization:**
   Comprehensive characterization of feedstock materials and DED samples through various techniques, including EDX, SEM, and EBSD.
3. **In-situ Measurements:**
   Execution of in-situ synchrotron and neutron measurements to capture real-time data on phase kinetics, stress and strain evolution and thermal dynamics during the deposition process.
4. **Data Analysis:**
   Comparison of collected data to identify correlations between process parameters, microstructural characteristics, and mechanical properties.



System Interface Planning

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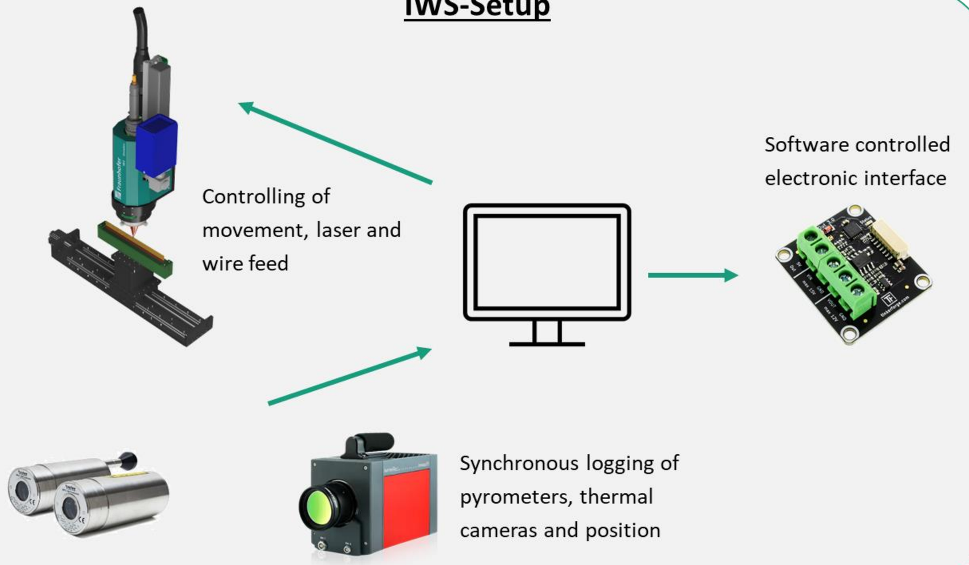
System Interface Planning:

IWS-Setup

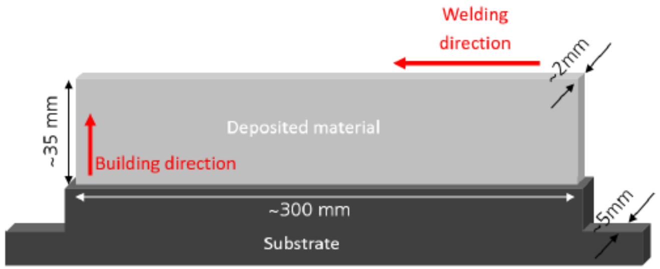


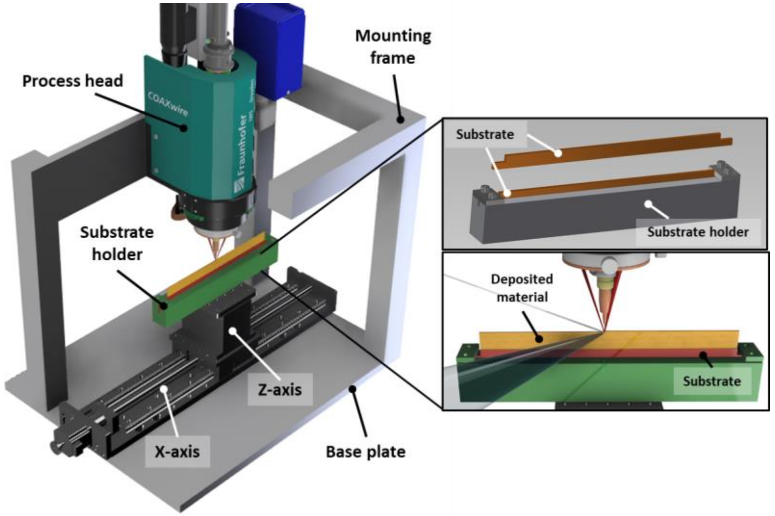
ESRF:

- „Laser Start“ trigger (3,3V HIGH)
- Pyrometer temperature 0-10 V

ILL:

- „Laser Start“ trigger (3,3V HIGH)
- Encoder signal for x-position
- Z-position form distance sensor





Process initialization
- Switch laser ready
- Prepare sensor system
- Setup wire feeder
- Setup motion systems

Process start
- Start wire feeder
- Start motion system
- Start Laser-Timer (wire startup phase)

Laser Start after Timer expiration
- Set Trigger signal HIGH
- Start Laser
- Storage of the trigger time
- Start of the sensor systems

End of process
- **Set Trigger LOW**
- Deactivate all Systems

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Material Characterization

TN 2

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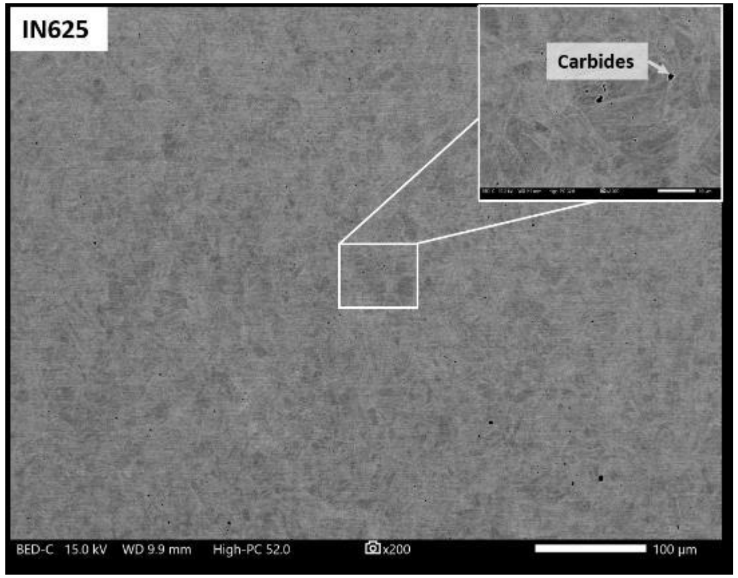
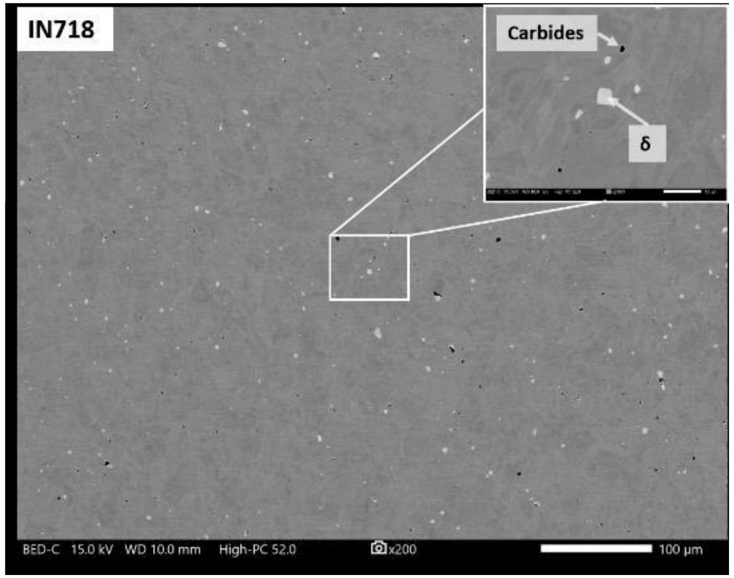
Material Characterization

Feed Stock Characterization
- Quality of IN718 and IN625 wires was confirmed through micrographs and EDX analysis.
- No significant contamination was detected.

Measured and nominal composition of the IN718 and IN625 wire:

Measured in wt-%	Ni	Fe	Cr	Mo	Nb	Ti	Al	Si
IN718	52,2	20,2	17,9	2,9	5,3	0,9	0,5	0,1
IN625	64,8	0,3	22,6	8,4	3,6	0,2	-	-
Nominal in wt.-%								
IN718	53	19,9	17,5	3	5	1	0,5	<0,1
IN625	61,1	<4	21	9	3,4	-	-	1





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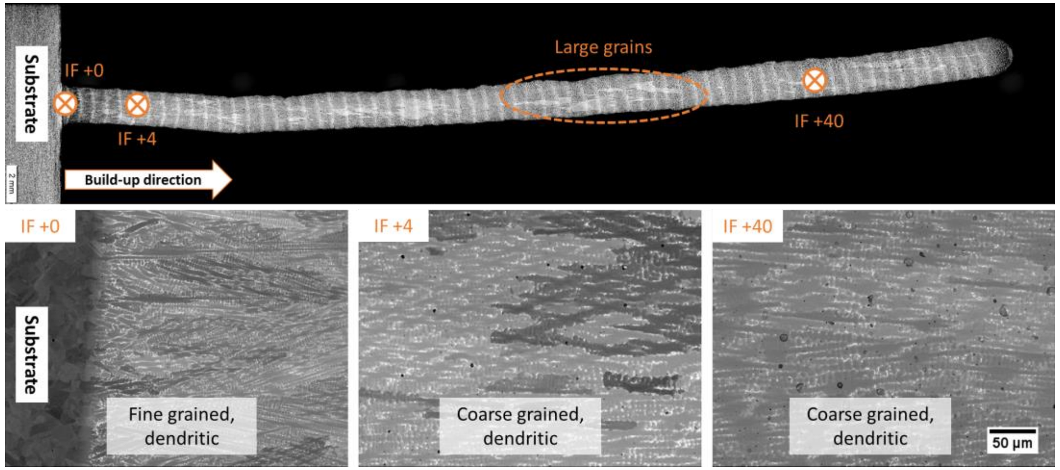
Material Characterization

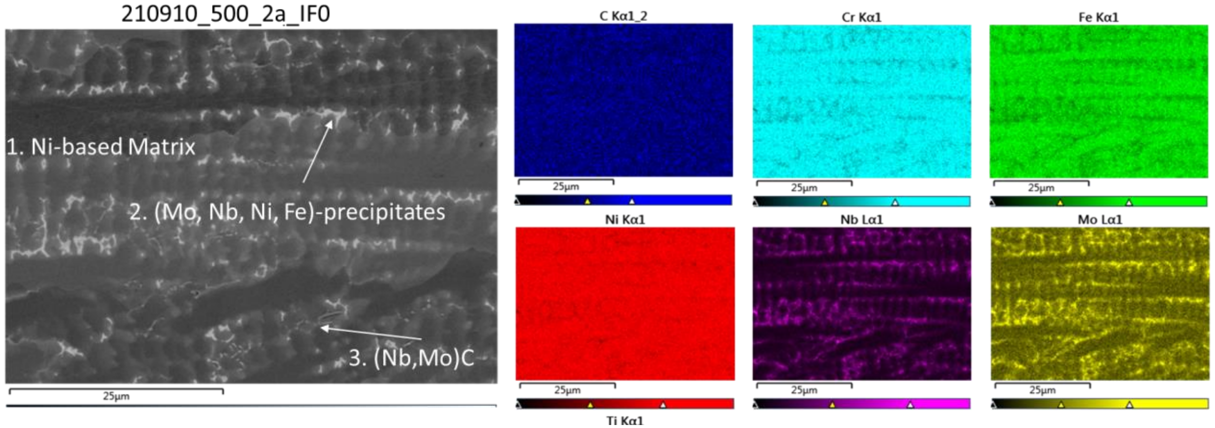
Preliminary Study – Sample Characterization

Light Microscopy (LM) and Scanning Electron Imaging (SEM)



Energy-dispersive X-ray spectroscopy

- Confirmed the presence of a Ni-rich matrix with Nb-rich carbides and interdendritic precipitates.



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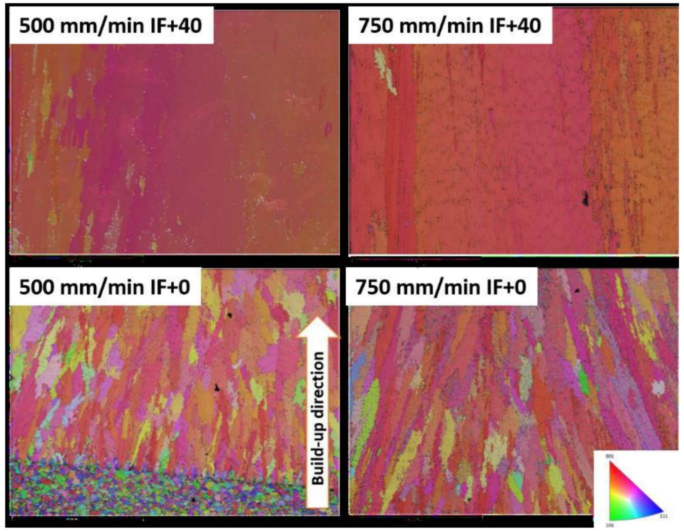
Electron Backscatter Diffraction (EBSD)

- Dendritic structures and a strong <100> fiber texture
- No significant differences in microstructure between varying welding speeds



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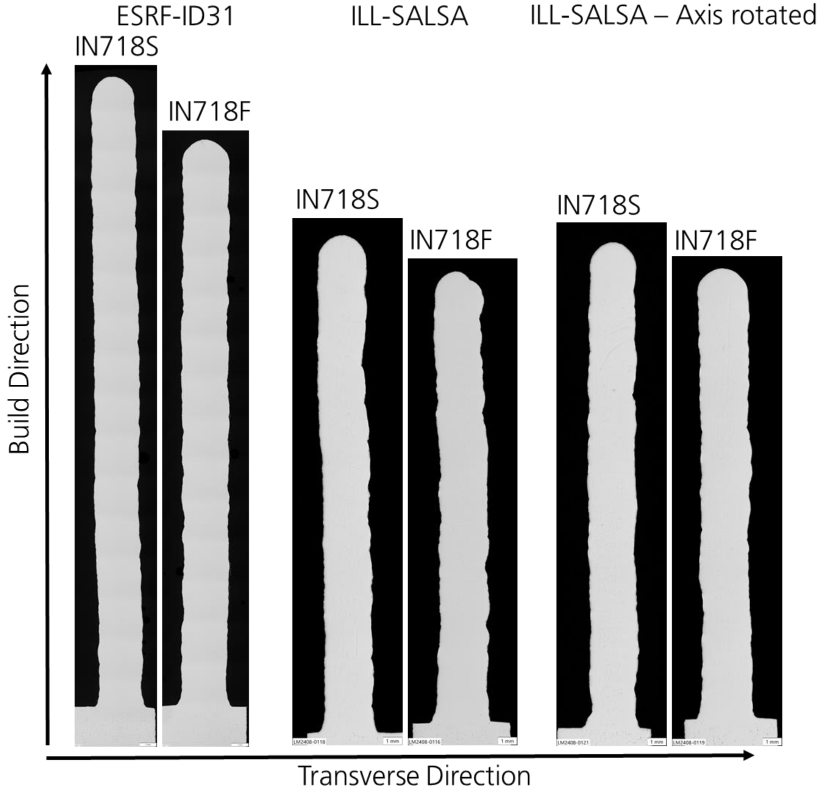
Material Characterization

ESRF and ILL – Sample Characterization

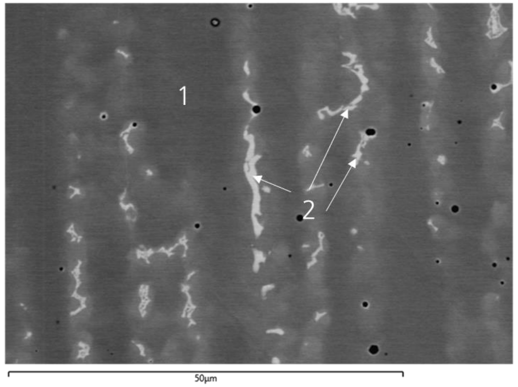
LM

- Irregularities in track shape observed in all IN718 samples.
- IN718S had greater build heights than IN718F.
- No larger pores or welding defects found.

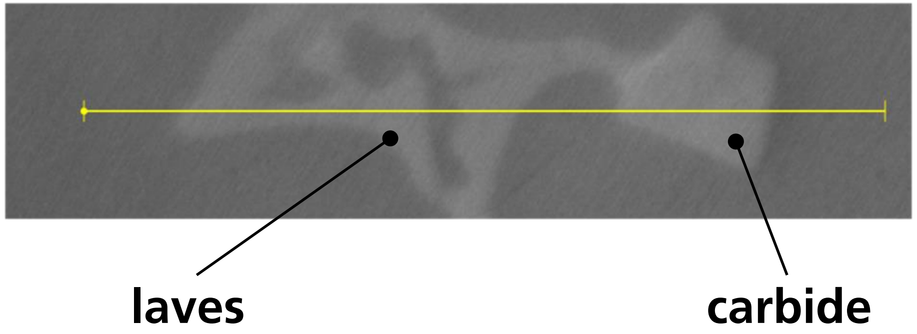


EDX and SEM

- Ni-based matrix with Mo/Nb-rich precipitates
- No distinct carbide phase identifiable in mapping
- EDX Line scans revealed joined appearance of laves and carbide phase



1. Ni-based Matrix as primary phase
2. Mo-, Nb-rich precipitates with reduced Fe and Cr content



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Material Characterization

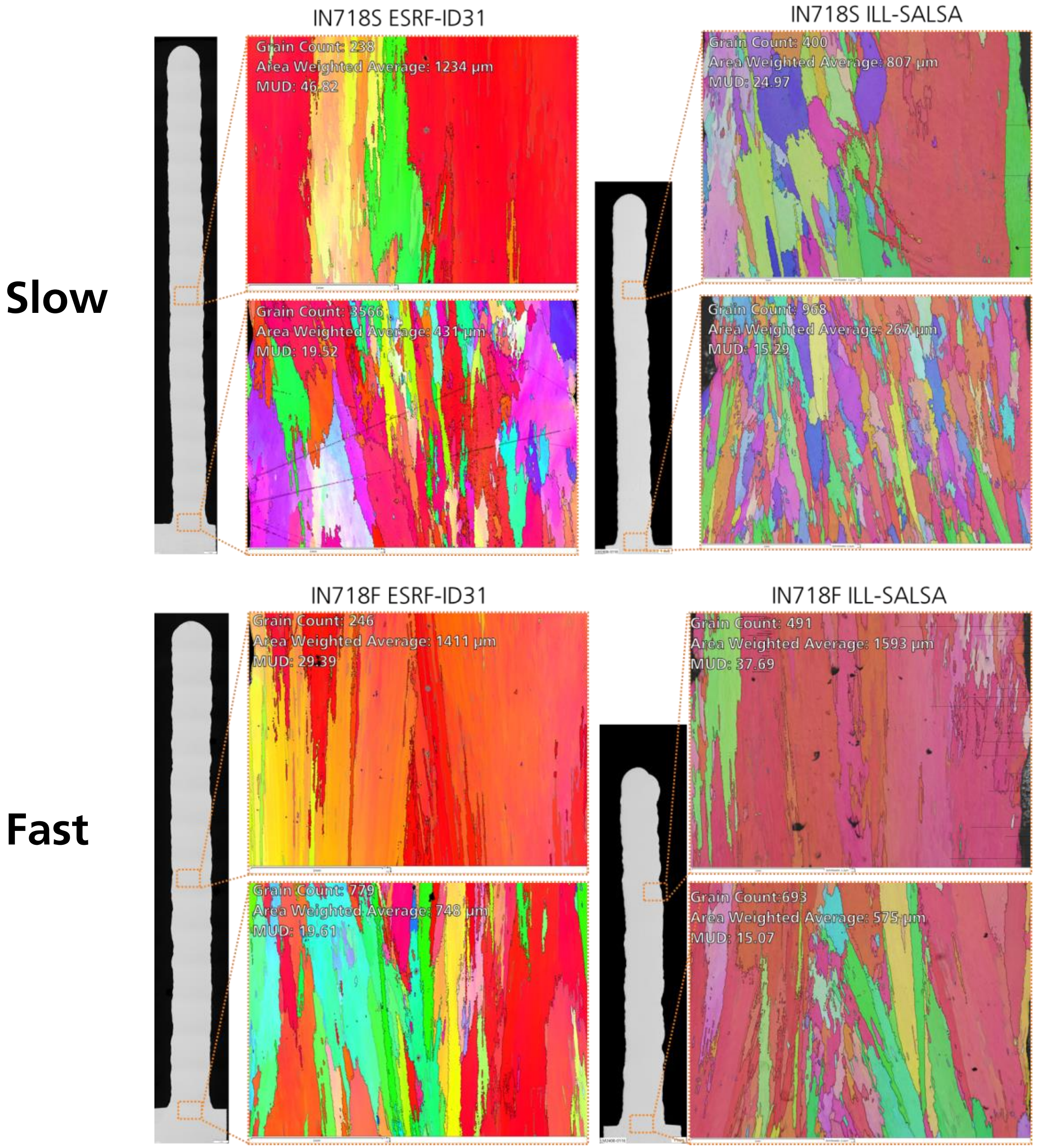
TN 6

ESRF and ILL – Sample Characterization

EBSD

Comparing parameter sets across locations

- Dendritic structure and large columnar grains in build direction
- No qualitative difference in the grain structure
- Higher grain counts and finder grains at substrate interface compared to bulk
- <100> orientation for all samples
- Significant differences in grain sizes and strength of texture but no clear trend



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Material Characterization

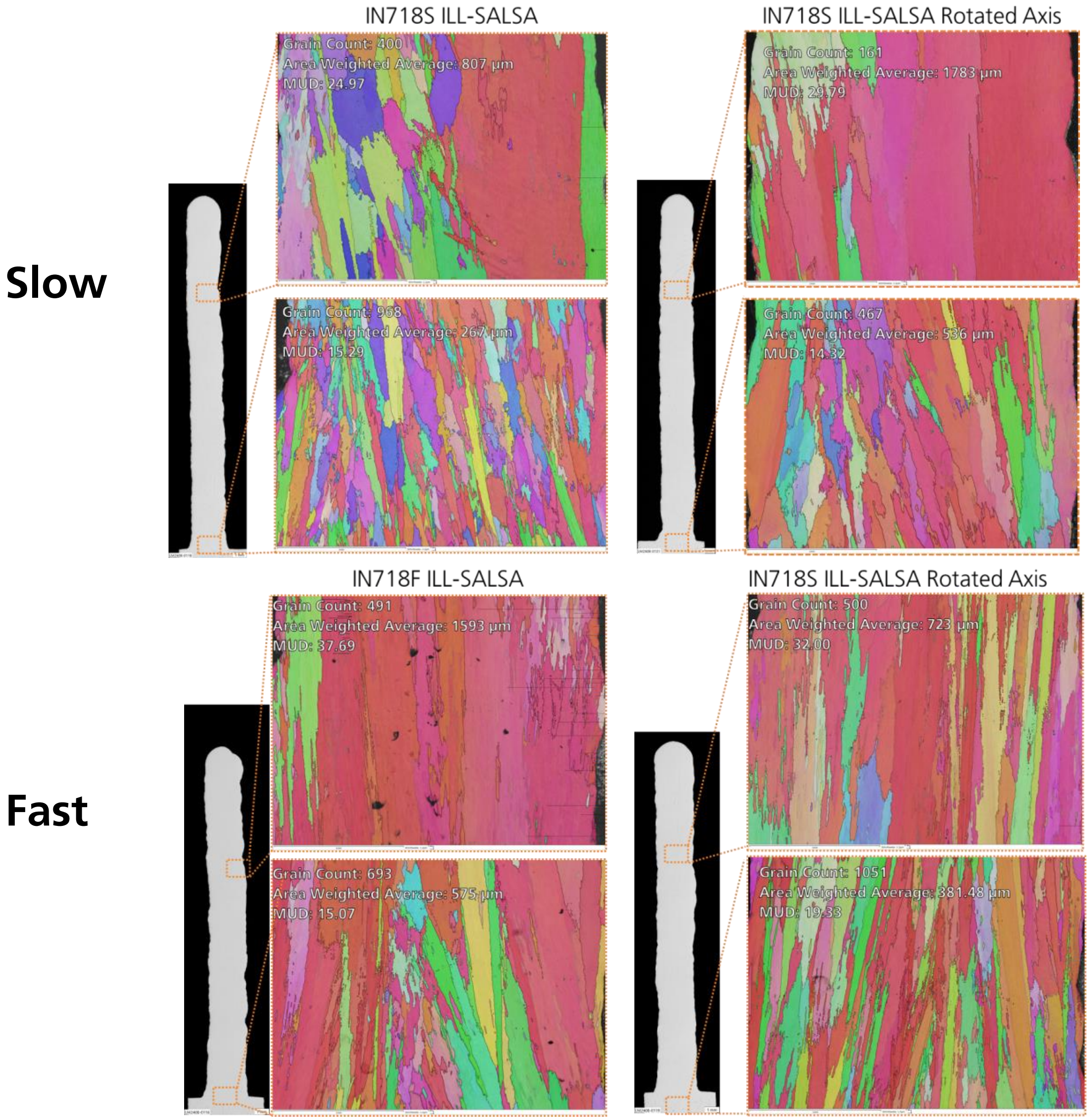
TN 6

ESRF and ILL – Sample Characterization

EBSD

Comparing the influence of axis rotation

- No qualitative difference in the grain structure
- Rotated axis: Larger grains in IN718S and finer grains in IN718F
- Quantitative difference but inconclusive results



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In-situ Measurements

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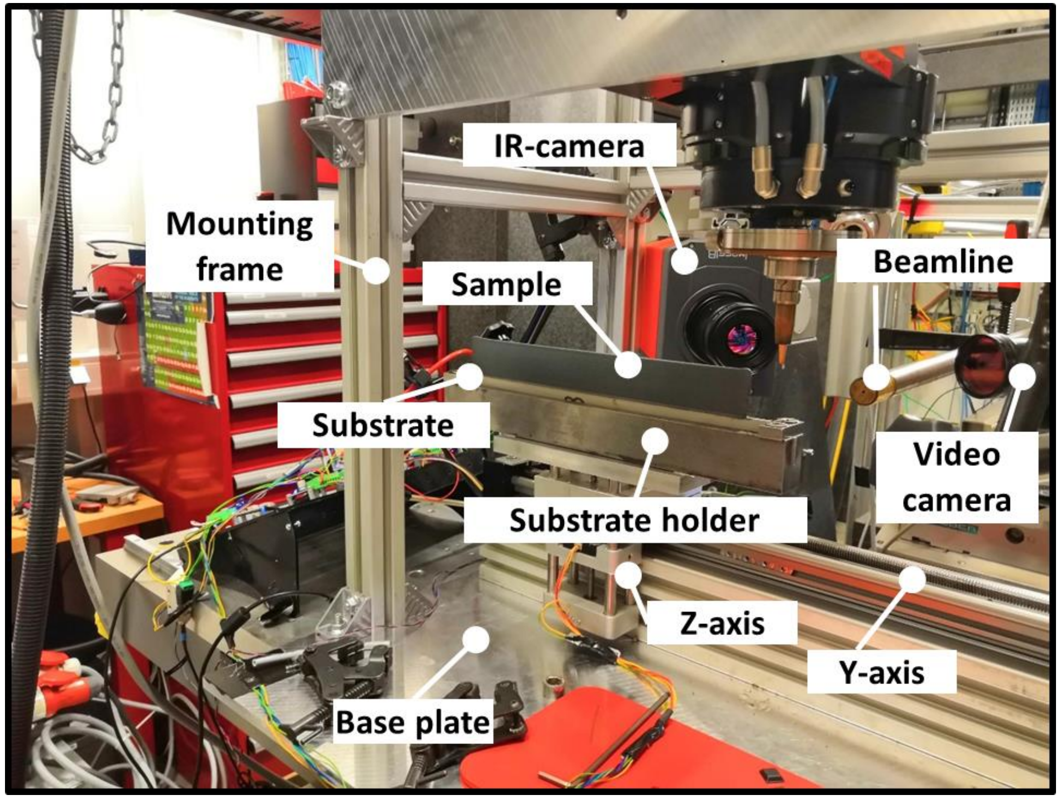
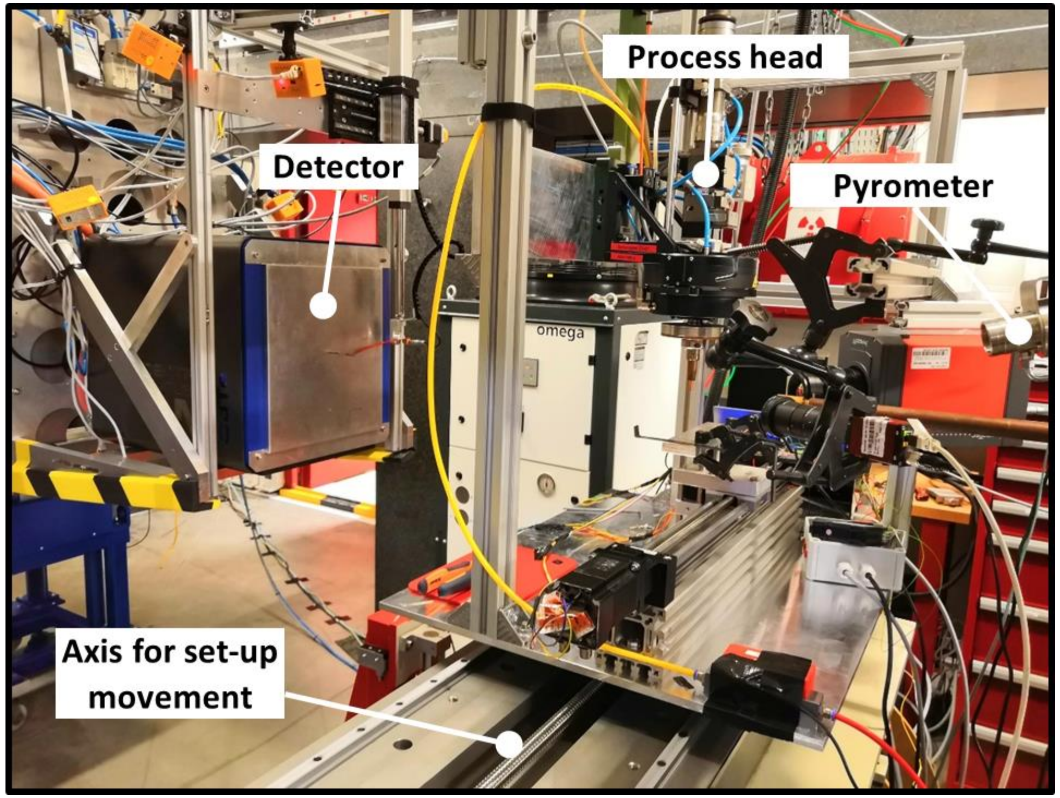
In-situ Measurements

TN 3

Set-up @ ESRF-ID31





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In-situ Measurements

Measuring strategy @ ESRF-ID31

Geometry:

- Wall → 300mm x 30mm x 2mm
- 60 layers total

Parameters:

- Slow: 500 mm/min, 1000 W
- Fast: 1000 mm/min, 1875 W
- 3 samples per parameter and material

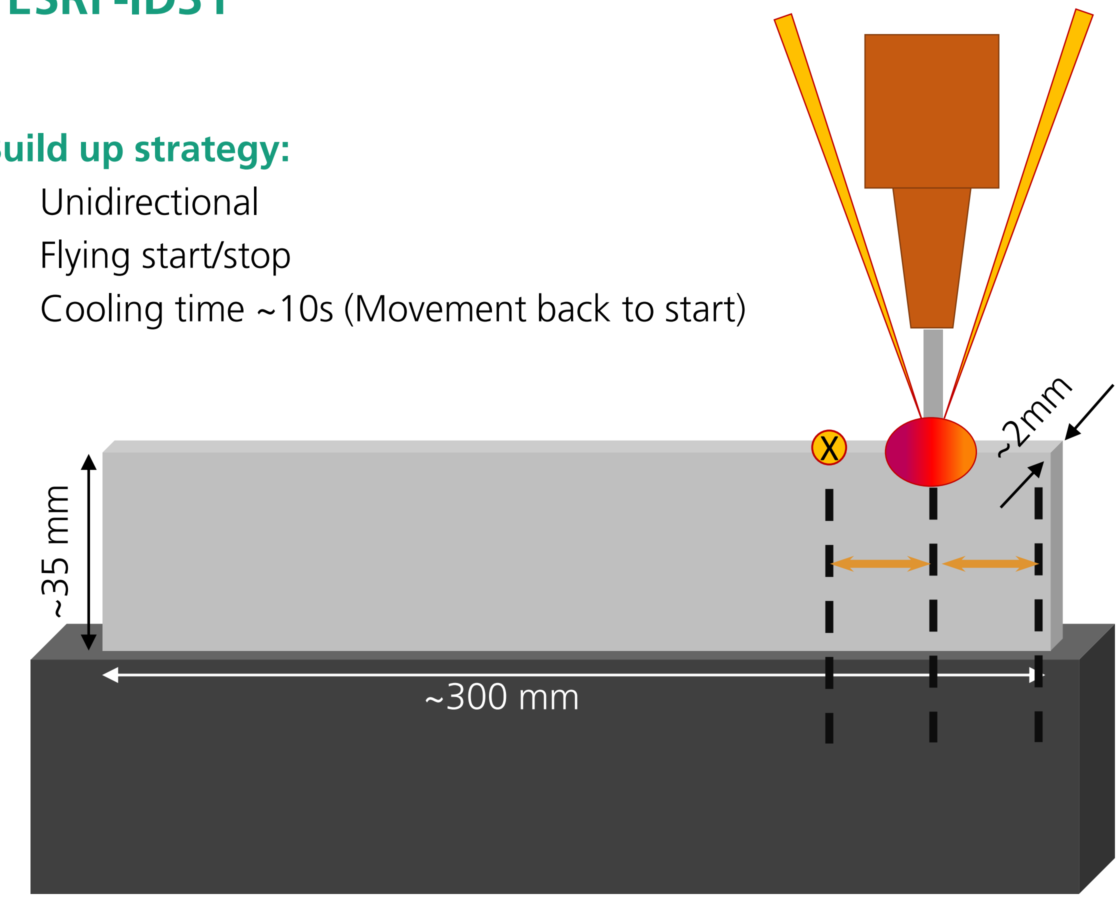
→ **12 Samples**

Build up strategy:

- Unidirectional
- Flying start/stop
- Cooling time ~10s (Movement back to start)

Ⓧ Position of x-Ray (changed by moving whole set-up)

- X-ray follows deposition layer for 20 layers (0-15mm built height)
- Scanning in x-direction +/-20mm around meltpool



→ **"Dynamic" region, information on solidification and phase kinetics**

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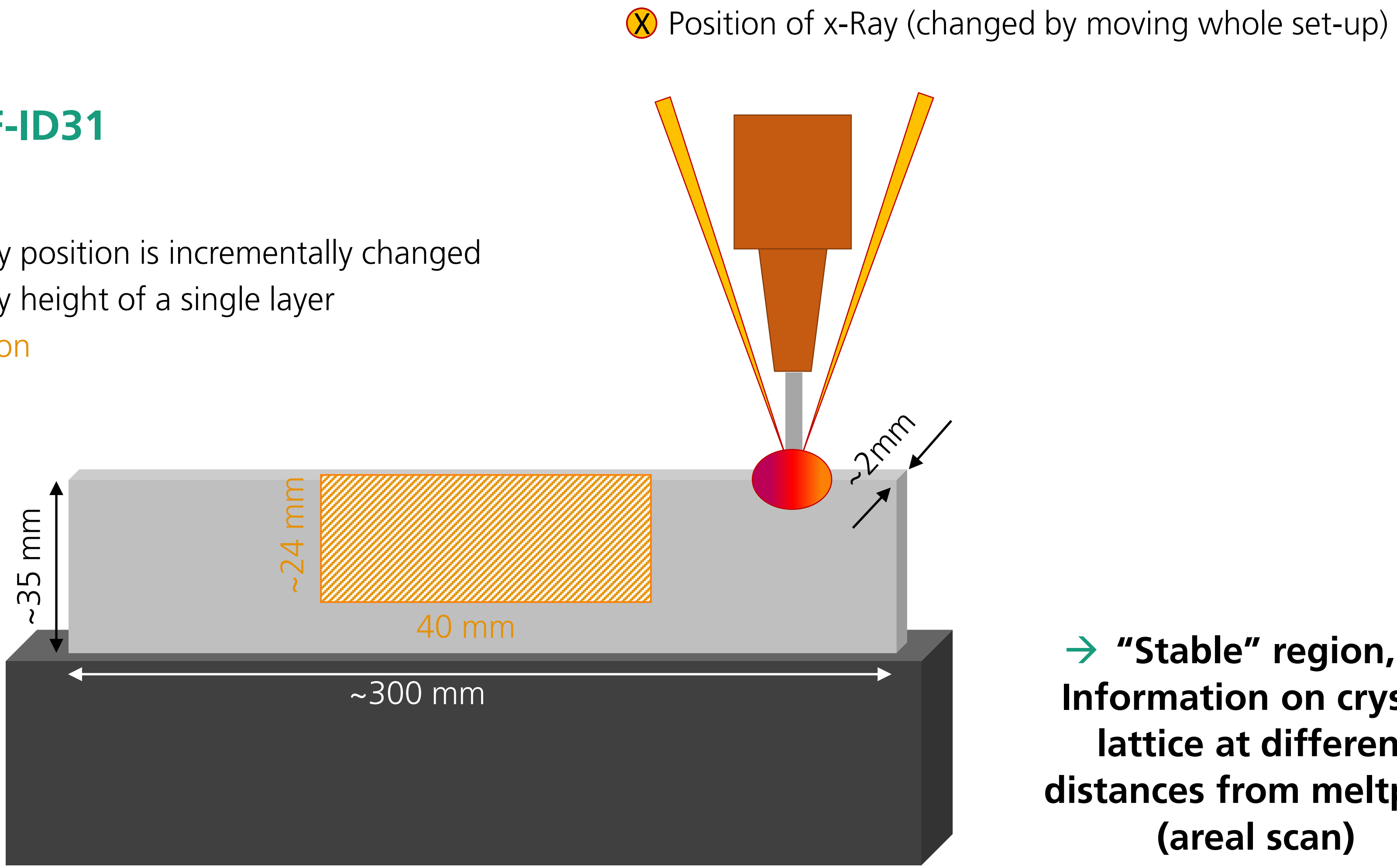
In-situ Measurements

Measuring strategy @ ESRF-ID31

- After 20 layers (stable regime) x-ray position is incrementally changed
- X-Ray pos. is moved downwards by height of a single layer
- 40x24 mm area scan in stable region



→ **"Stable" region, Information on crystal lattice at different distances from meltpool (areal scan)**

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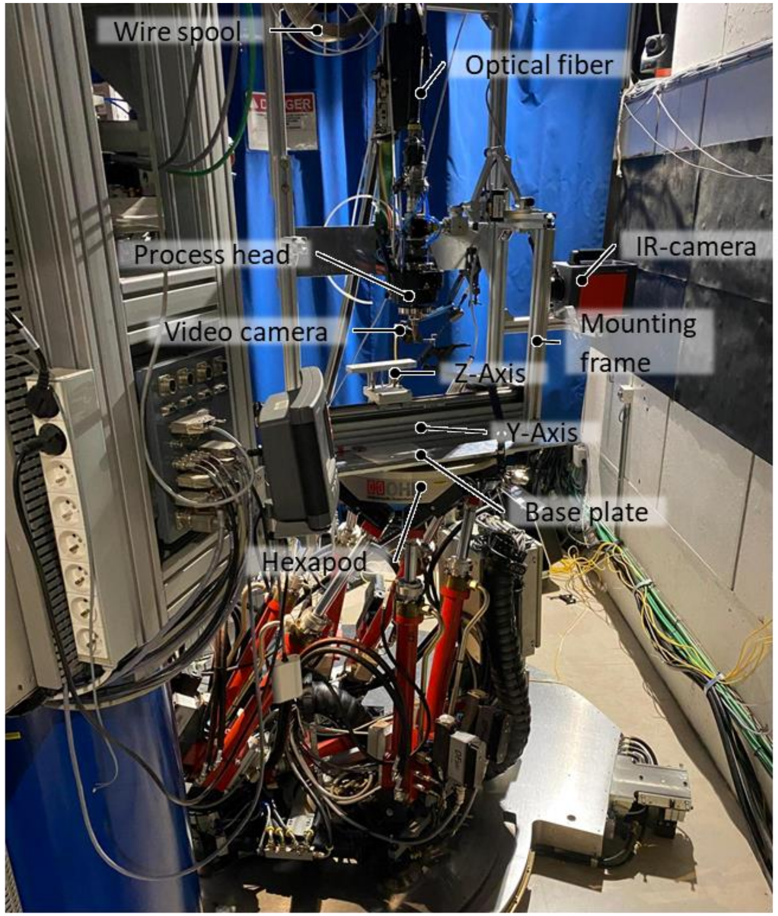
In-situ Measurements

TN 5

Set-up @ ILL-SALSA



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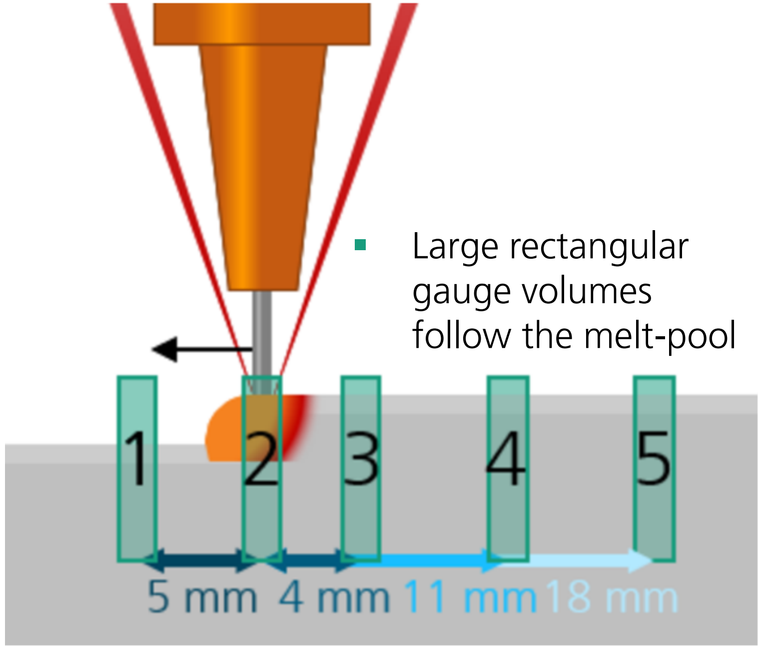
In-situ Measurements

TN 5

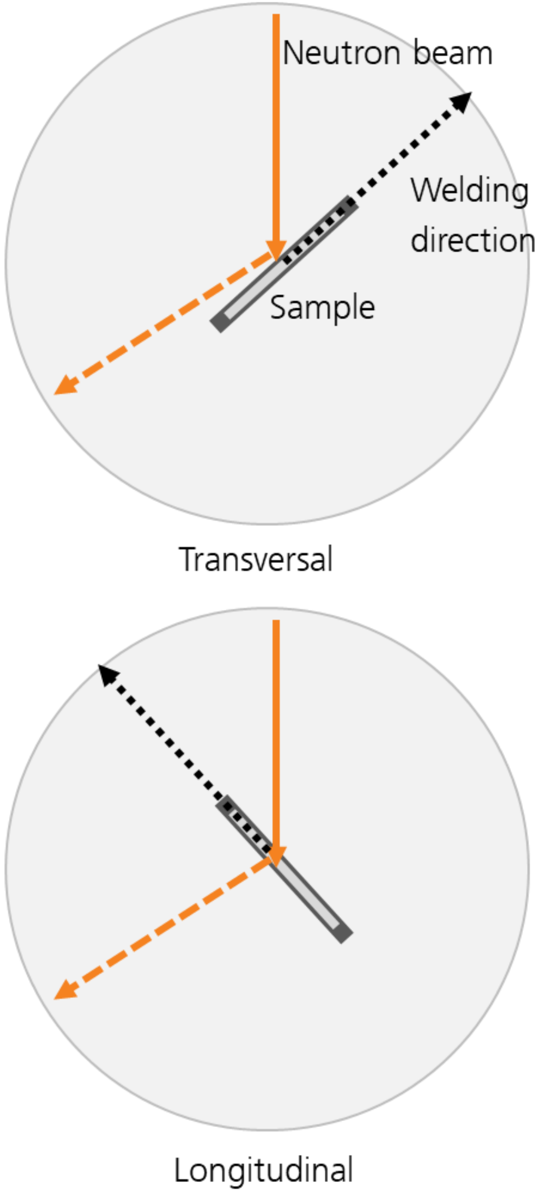
Measuring strategy @ ILL-SALSA

Geometry:
- Wall → 300mm x 30mm x 2mm
- 40 layers total

Build up strategy:
- Unidirectional
- Flying start/stop
- Cooling time ~10s (Movement back to start)

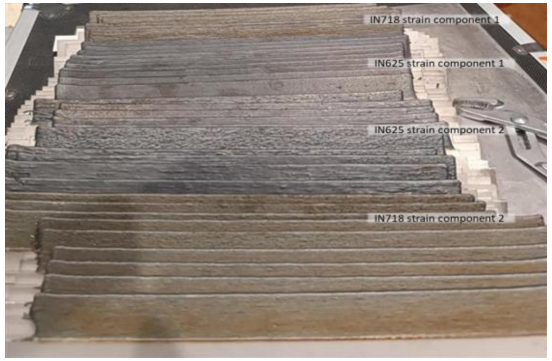


- Large rectangular gauge volumes follow the melt-pool



Measuring Strategy:
- 2 Sample orientations
- 2 Materials
- 2 Parameters sets
- 5 Positions

→
40 Samples
(~57 hours of measuring time at ILL-SALSA)



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Analysis

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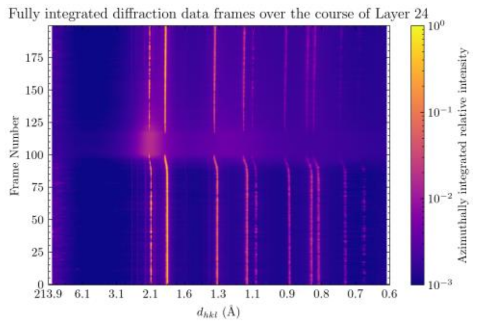
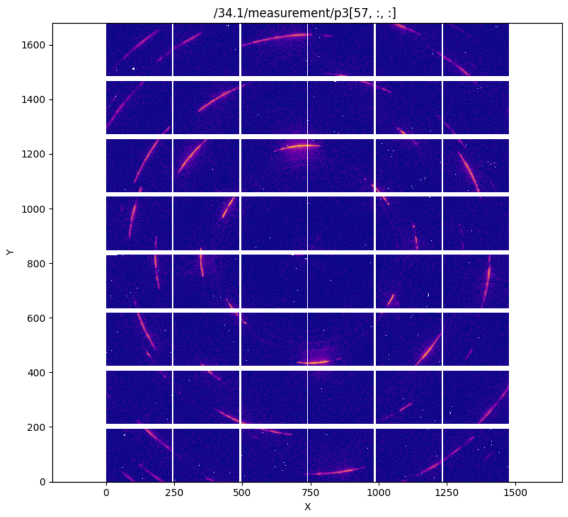
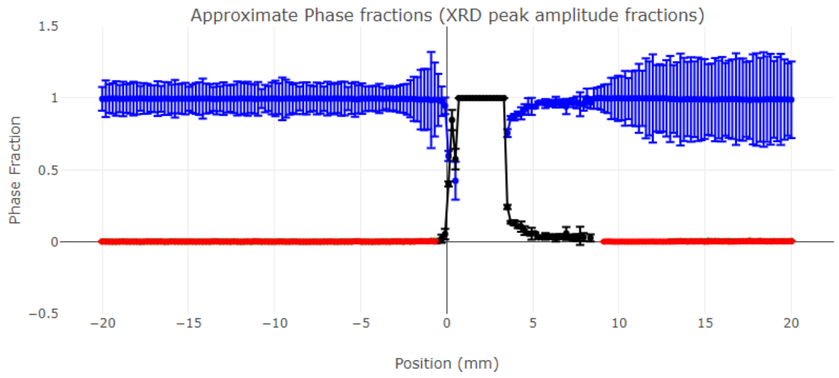
- ESRF – Phase Analysis



Pre Processed Data → Analysis → Qualitative Phase Evolution / Semi Quantitative Phase Evolution



- Peak identification
- Data extraction
- Peak Fitting (xrdfit)
- Filtering (Fit Std. Err., Height rel to Bg., FWHM)
- Peak averaging
- Data visualization



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Analysis

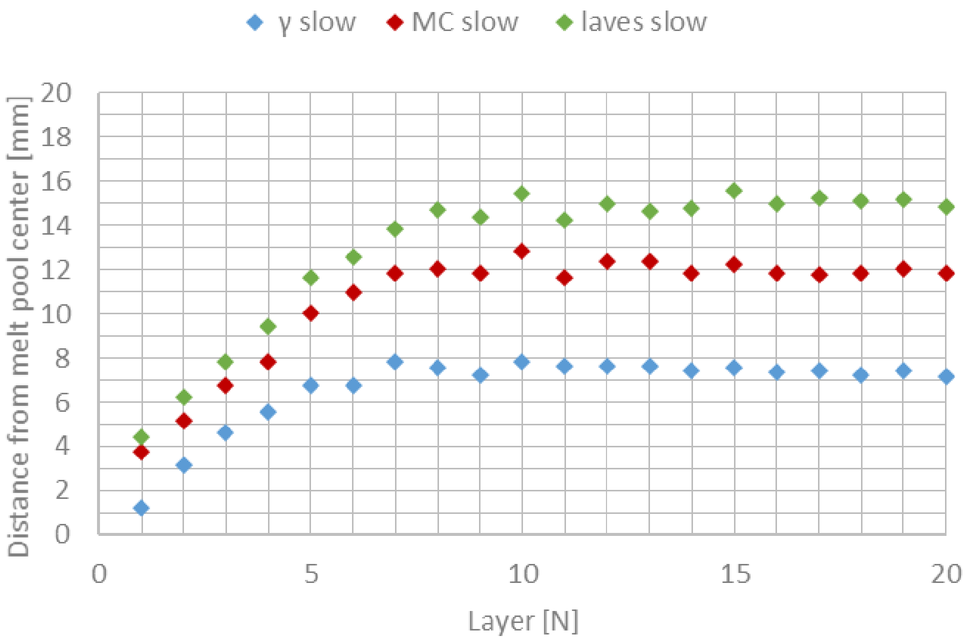
TN 4

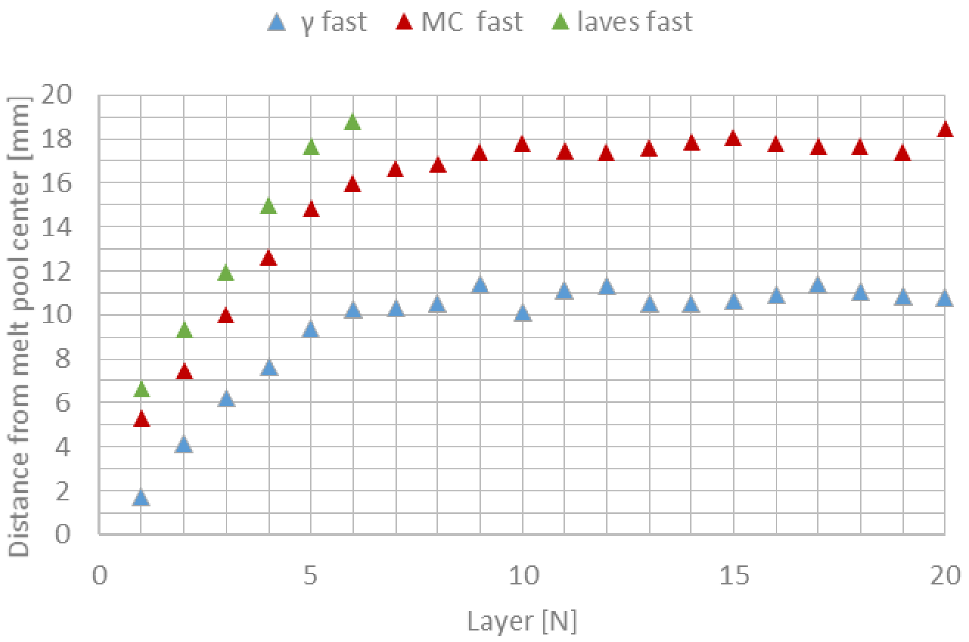
In-situ Qualitative Phase Analysis

- **Space** resolved phase evolution for layers 1 to 20 (when is a phase first detected) dependent on the parameter set

Findings:

- Identified phases include γ-phase, carbide phase, and laves phase.
- γ-phase appeared first during solidification, followed by MC and Laves phases.
- Distance from melt pool center to solidification front increased with each layer until stabilizing around layer 7
- Fast parameter set results in greater distance from the melt pool before solidification occurs
- IN718 displayed a more pronounced influence of welding speed on γ-phase evolution compared to IN625.





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Analysis

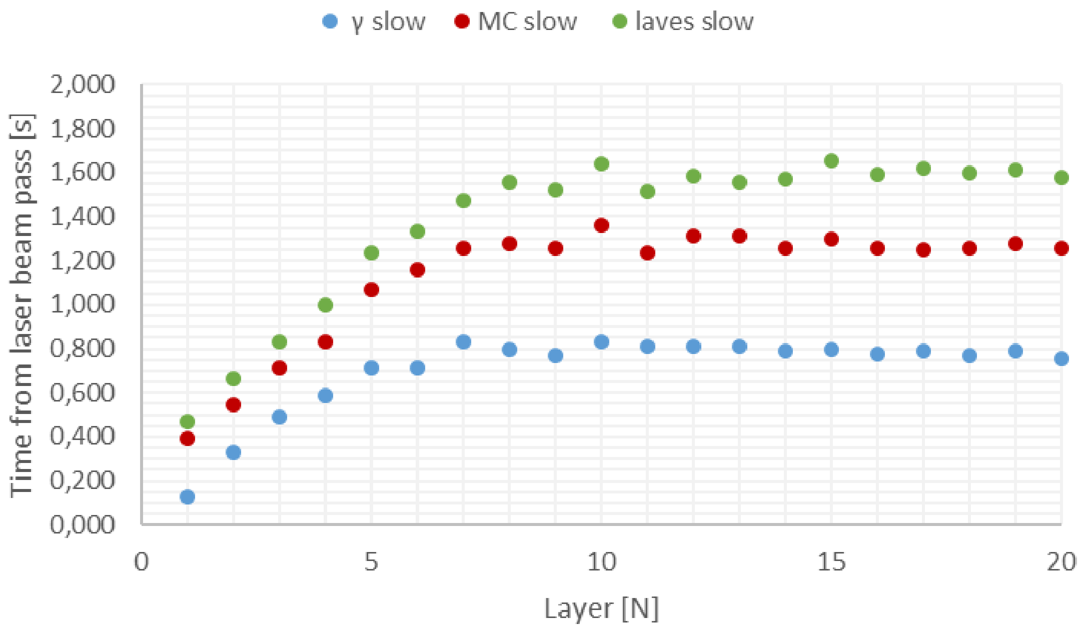
TN 4

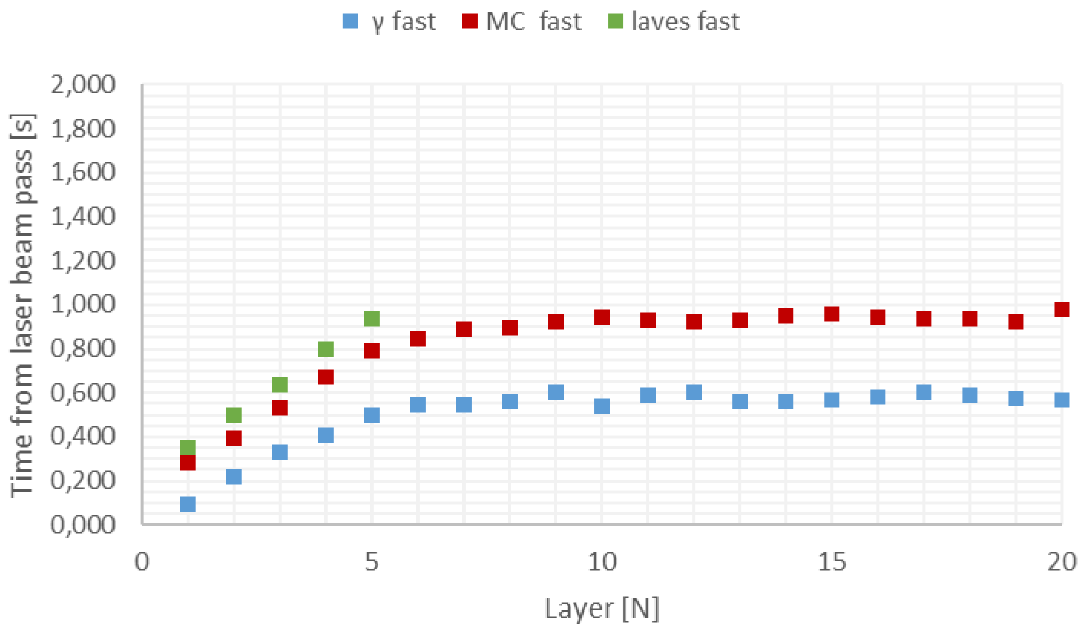
In-situ Qualitative Phase Analysis

- **Time** resolved phase evolution for layers 1 to 20 (when is a phase first detected) dependent on the parameter set

Main Findings:

- γ-phase appeared first during solidification, followed by MC and Laves phases.
- time from laser beam passing to solidification front increases with each layer until stabilizing around layer 7
- Fast parameter set results in faster solidification after laser beam passing





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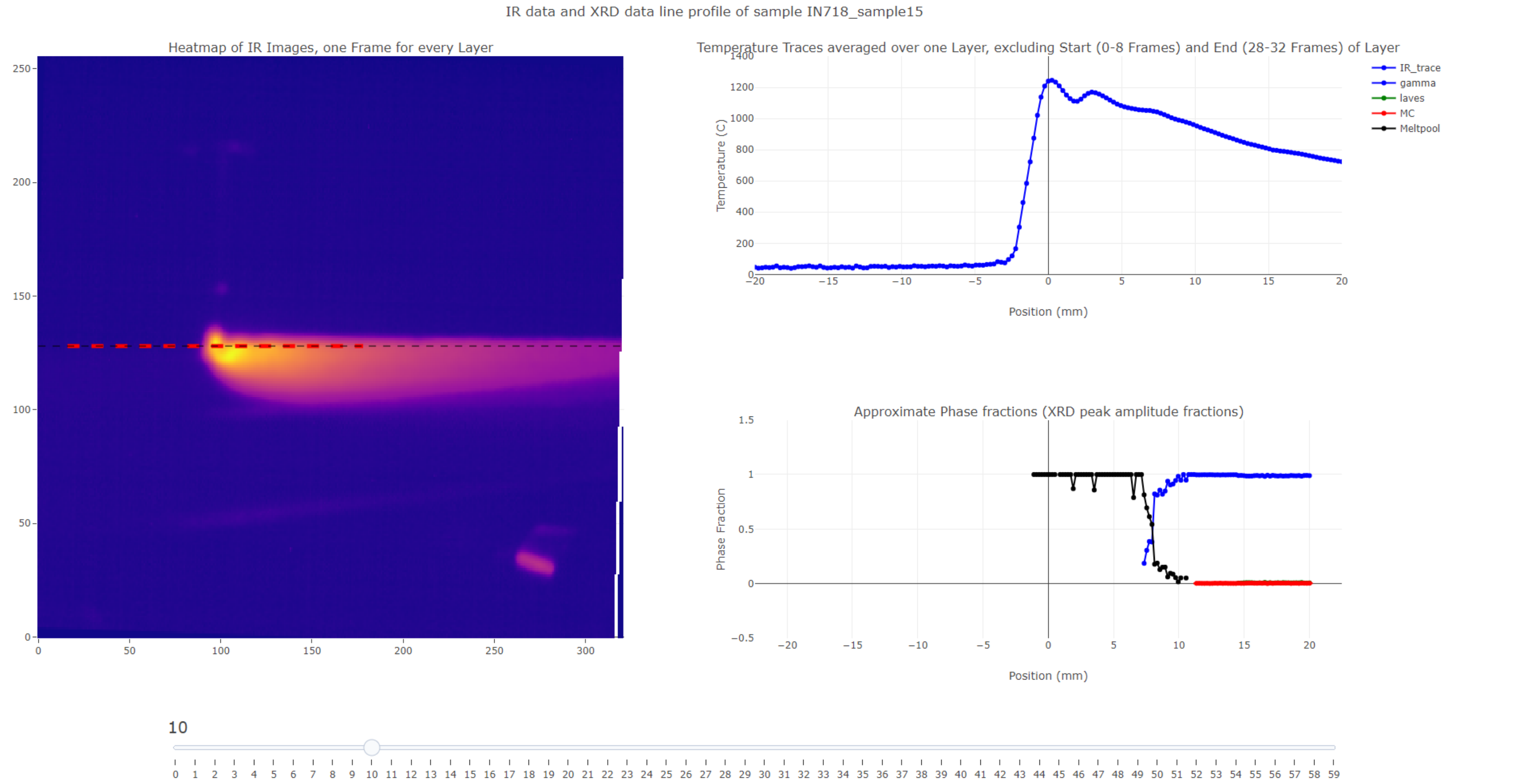
Analysis

TN 4

+ Appendix

In-situ Semi Quantitative Phase Analysis

- Phase fractions provide rough estimates due to strong texture in samples, limiting direct comparison between phases
- Space resolved, semi-quantitative phase evolution over the entire measurement linked to temperature data from the IR-camera
- Available as interactive graphical user interface for data exploration



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Analysis

Ex-situ Semi Quantitative Phase Analysis

- Resulting phase fractions provide rough estimates due to strong texture in samples, limiting direct comparison between phases.
- Space resolved phase analysis over the entire build height
- Also Available as interactive graphical user interface for data exploration

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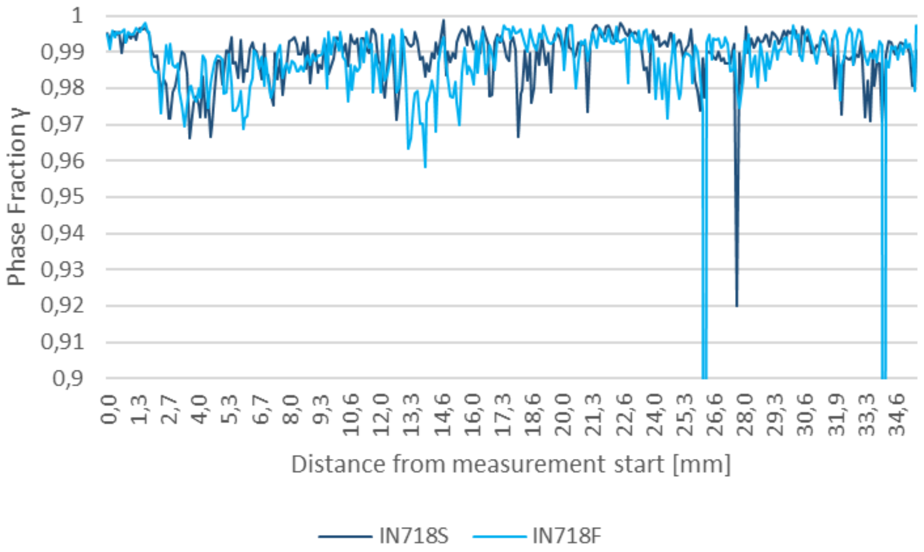

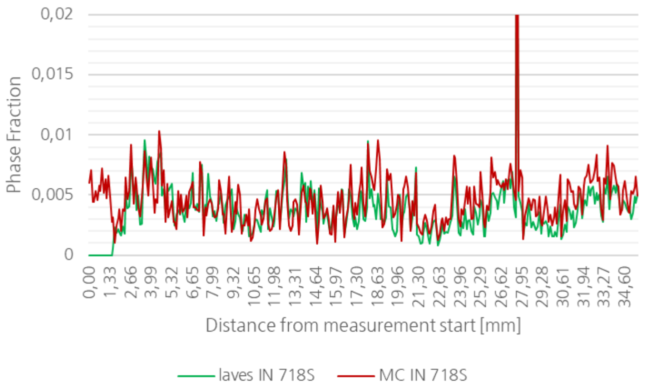

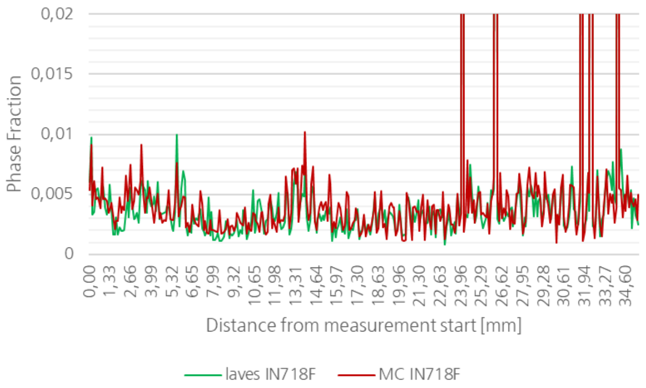

Main Findings

- IN718S: Average γ phase fraction is 0.9888
- IN718F: Average γ phase fraction is 0.9855
- Strong correlation between the Laves and MC phase fraction evolutions, with coefficients of 0.881 for IN718S and 0.972 for IN718F.

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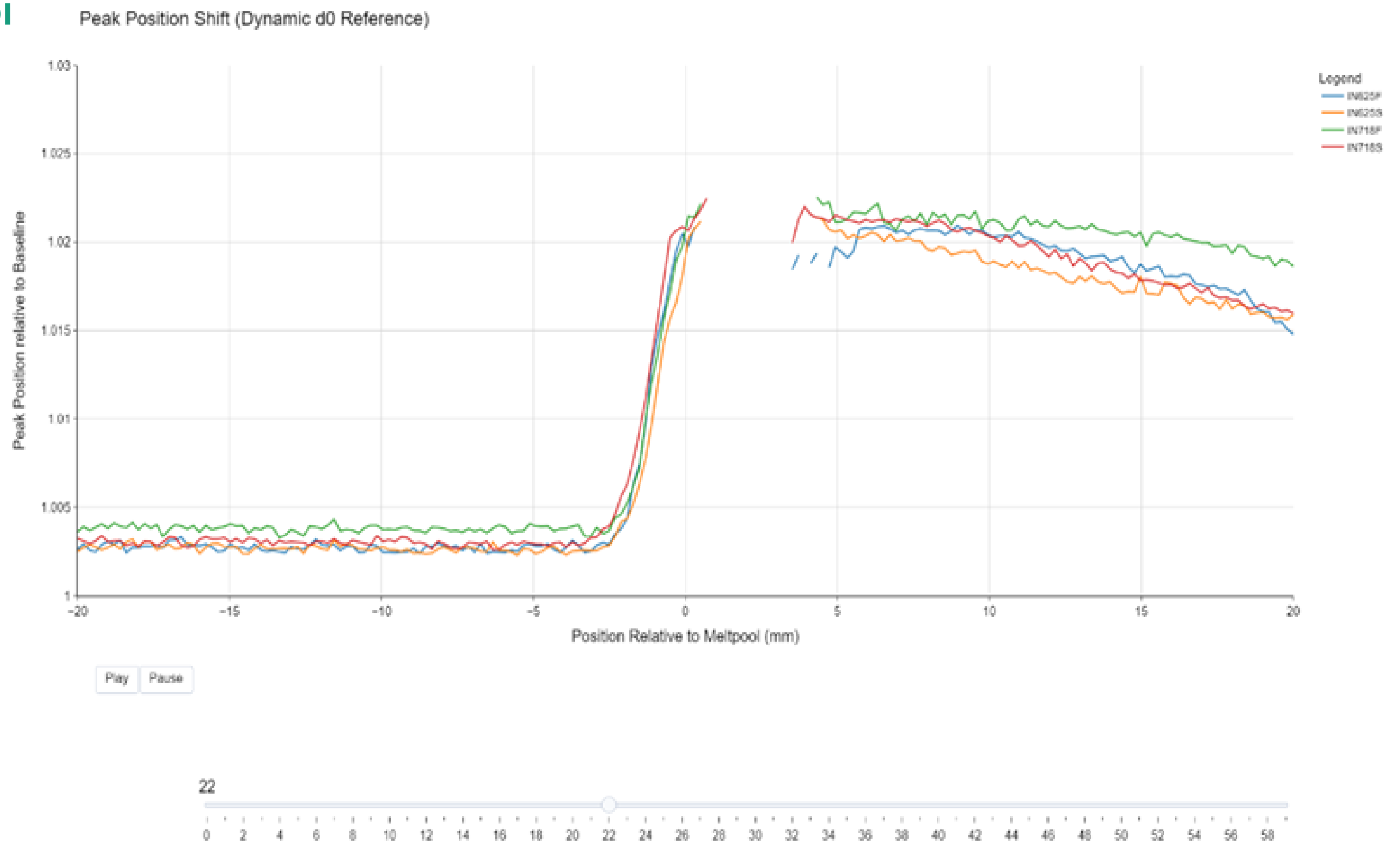
Analysis

TN 4

+ Appendix

Peak Shift Caused by Temperature Variatio

- Peak center as one of the fit parameters can be used to gain information on the expansion of the crystal lattice due to temperature
- Relative peak shift as a function of position relative to the melt pool center for every parameter set for both IN625 and IN718
- Available as interactive graphical user interface for data exploration



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Analysis

Ex-situ Strain Analysis

Due to the pronounced texture and significant grain size in the deposited material, calculations were feasible only up to approximately 5 mm from the interface

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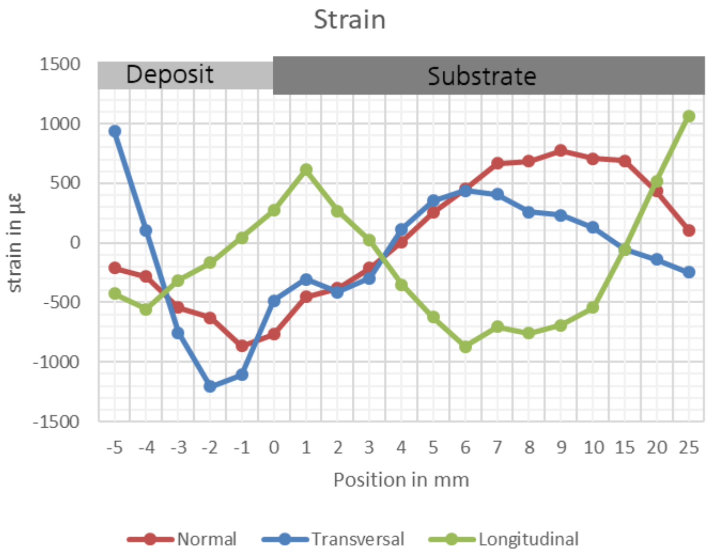
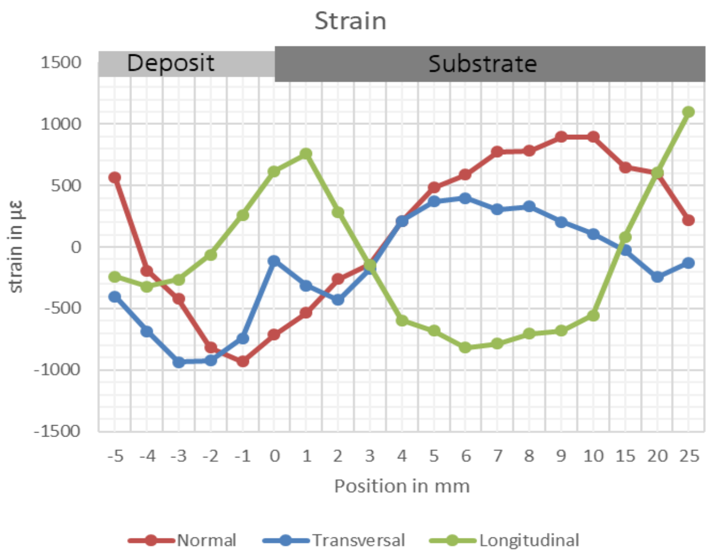
Slow



Fast



Strain component	Normal	Transversal	Longitudinal
Correlation factor	0,95	0,69	0,97

	Average absolute strain value in με		
Component	Normal	Transversal	Longitudinal
IN718S	480,33	420,04	465,88
IN718F	561,22	370,85	502,77

Main Findings

- Fast parameter set exhibits higher average absolute values for both normal and longitudinal strains
- Lower average absolute values for transverse strain.
- Most significant difference in average absolute strain in normal component.
- Most significant difference in correlation in transversal component

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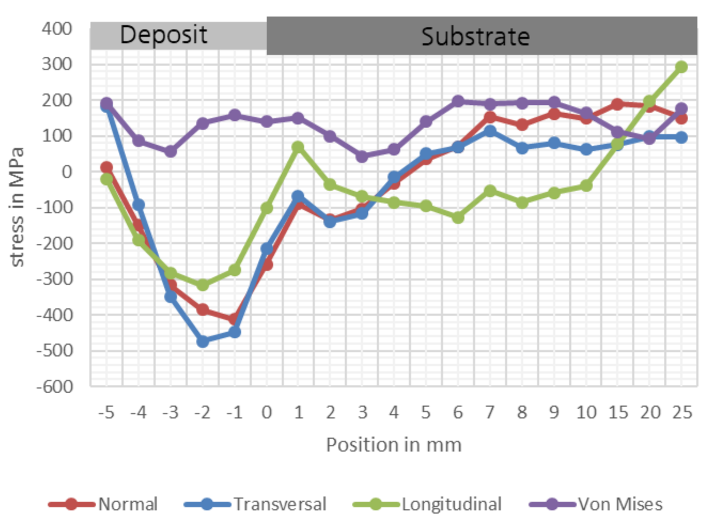
Analysis

Ex-situ Stress Analysis

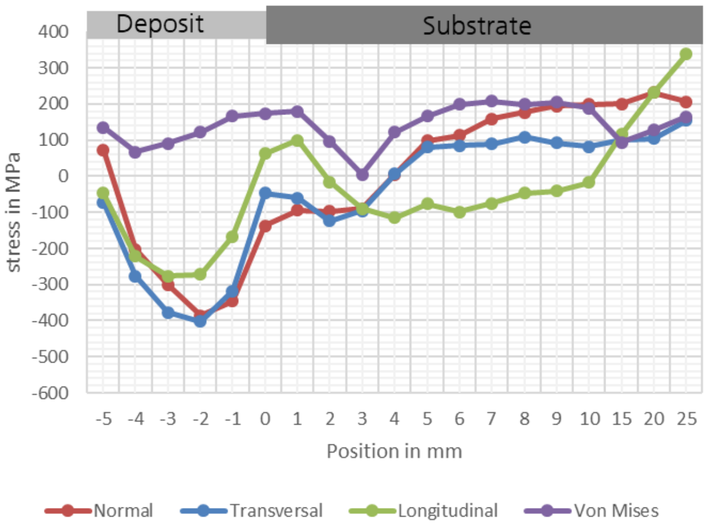
Due to the pronounced texture and significant grain size in the deposited material, calculations were feasible only up to approximately 5 mm from the interface

	Average absolute stress value in MPa			
Component	Normal	Transversal	Longitudinal	Von Mises
IN718S	164,21	148,18	130,29	135,94
IN718F	174,35	140,62	126,86	142,73

Slow



Fast



TN 5

Stress component	Normal	Transversal	Longitudinal	Von Mises
Correlation factor	0,98	0,88	0,95	0,86

Main Findings

- Fast parameter set exhibits higher average absolute values for both normal and Von Mises stress
- Lower average absolute values for both transverse transversal and longitudinal stress.
- Most significant difference in correlation in transversal component and von Mises stress

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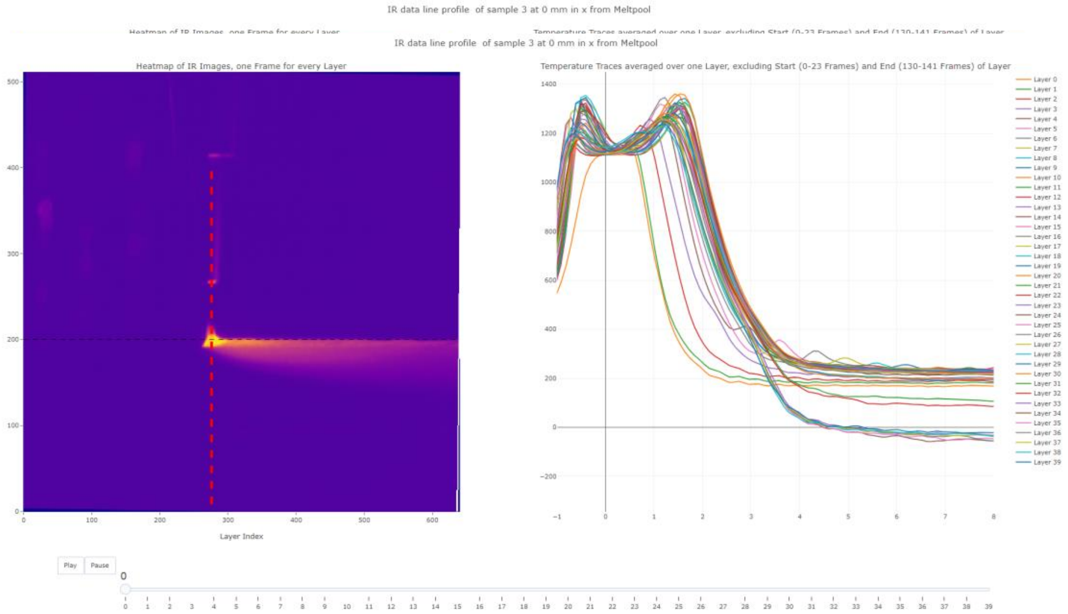
Analysis

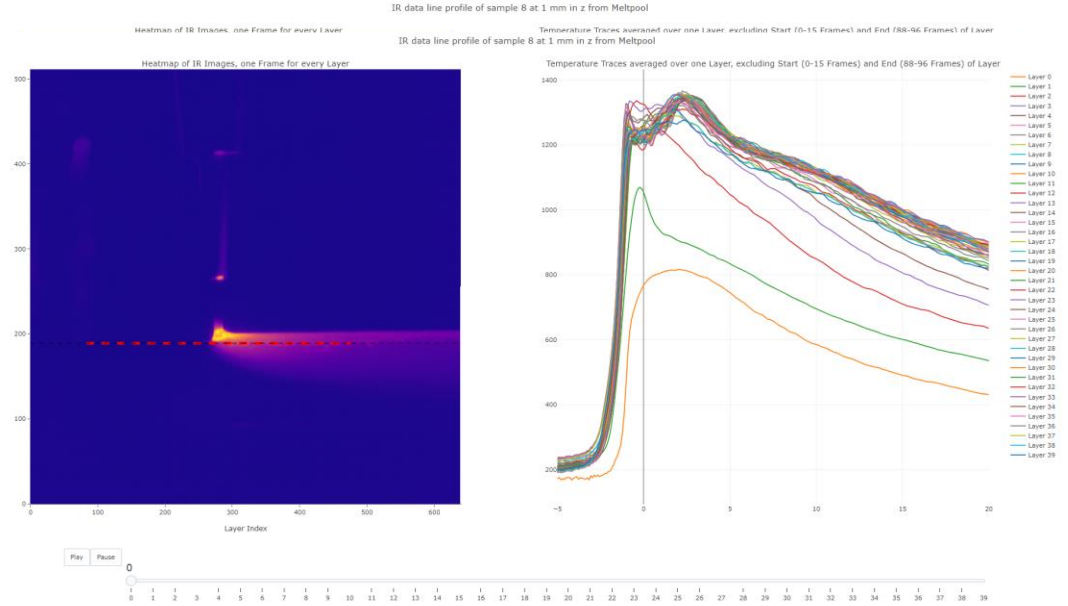
IR-camera data

- Data processed and made available as interactive graphical user interface for data exploration
- Different profiles and camera calibrations available
- Remaining uncertainties:
  - Automatic melt pool detection
  - Different emission coefficients
  - Inconsistent build height

Vertical



Horizontal



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Unachieved Objectives

In-situ neutron data analysis

Not completed due to unforeseen challenges such as:

- Delays in measurement campaigns Sinusoidal laser output due to ILL power-infrastructure
  → 1. failed campaign
  - Breaking Down of Laser power supply → 2. failed campaign
- Technical issues with data processing
  - Unexpectedly weak support from ESRF staff
  - Suboptimal sample microstructure for analysis
  - High data complexity

Comprehensive understanding of the dynamic strain evolution during DED Not achieved due to lack of in-situ neutron data analysis

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Summary: Main Results & Findings

Results

- Improved system and measuring setup
  - stable process regimes
  - synchronized data acquisition
  - temperature monitoring
- Successful in-situ synchrotron and neutron measurements.
- Ex-situ synchrotron and neutron measurements
- Characterization using LM, Micro Hardness Testing, SEM, EDX, EBSD
- Comprehensive, statistically validated dataset for a defined geometry with known boundary conditions for DED-LB/M of IN625 and IN718
- Key insights for potential future measuring campaigns

Findings

- Solidification kinetics and phase formation of IN625 and IN718 (in-situ synchrotron) are significantly influenced by process parameters
- Stress and Strain Distribution around the substrate-deposit interface (ex-situ neutron) are weakly impacted by process parameters
- Synchrotron results mostly supported by characterization and IR data
- Generally good process repeatability, biggest uncertainty is impact of axis rotation
- Process parameters significantly influence bulk material temperature

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Outlook

Possible scenario for in-situ neutron data analysis:

Research assistant + Master Thesis

- Physics student (or related field)
- 6 Months research assistant/internship + 6 months masters thesis
- Atleast 4 weeks @ ILL for collaboration with instrument scientists
- Assistance by ILL-scientists required for supervision and analysis

Exemplary time table and rough work plan for in-situ data analysis.

	Year	2025/2026											
Activity	Month	2	3	4	5	6	7	8	9	10	11	12	1
Scientific Assitant: Introduction to DED-LB/M wire. Introduction to the activites within the De-Risk activity Familarization with the data and the required software tools													
Master Student at ILL : 1 Month visit to ILL during reactor downtime. Data analysis in cooperation with ILL instrument scientists.													
Master Student at IWS Finalizing data analysis Writing of thesis and reports													