Operando synchrotron x-ray diffraction during metal additive manufacturing to de-convolute the complex nature of the solidification process

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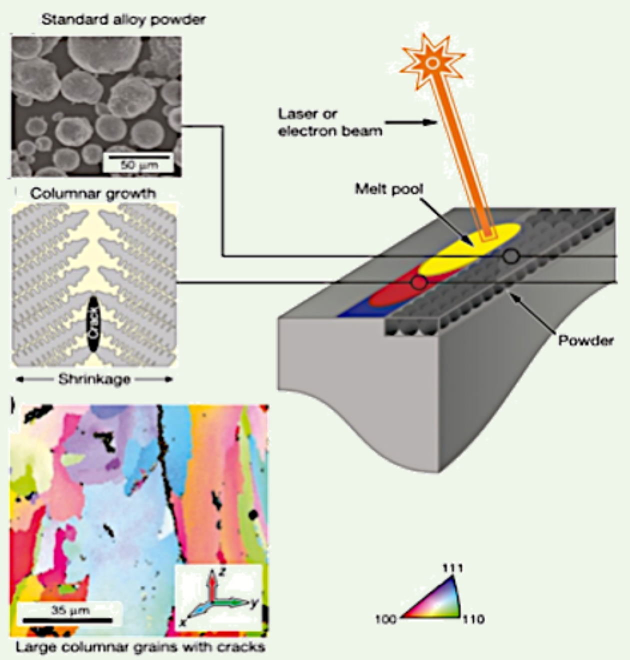
Gap in understanding the mechanisms of non-equilibrium solidification pathway



Common techniques used to study solidification in metal AM

Solidification Front velocity

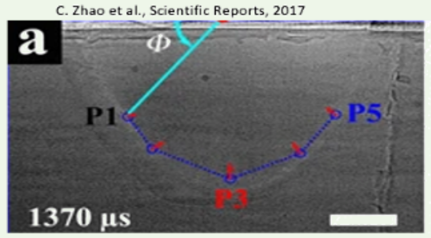
Cooling Rate

$$R = \frac{1}{G}\frac{\partial T}{\partial t} = \frac{1}{\sqrt{\left(\frac{\partial T}{\partial \xi}\right)^2 + \left(\frac{\partial T}{\partial z}\right)^2}}\frac{\partial T}{\partial t}$$

Thermal Gradient

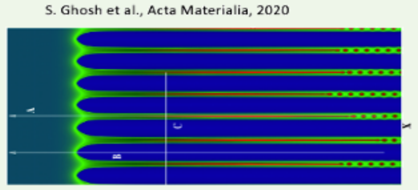
Analytical Models

C. Zhao et al., Scientific Reports, 2017



Synchrotron X-Ray Imaging

S. Ghosh et al., Acta Materialia, 2020



Phase Field Modelling

Why synchrotron diffraction?

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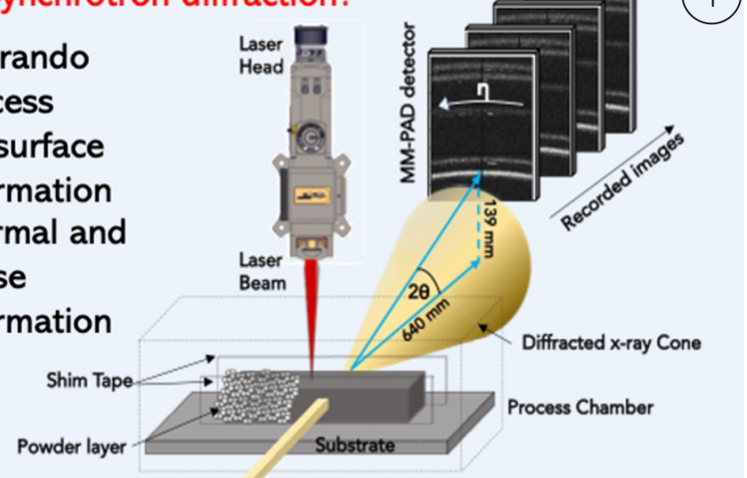
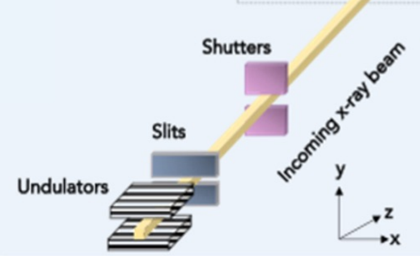
- ❖ Operando process
- ❖ Subsurface information
- ❖ Thermal and phase information

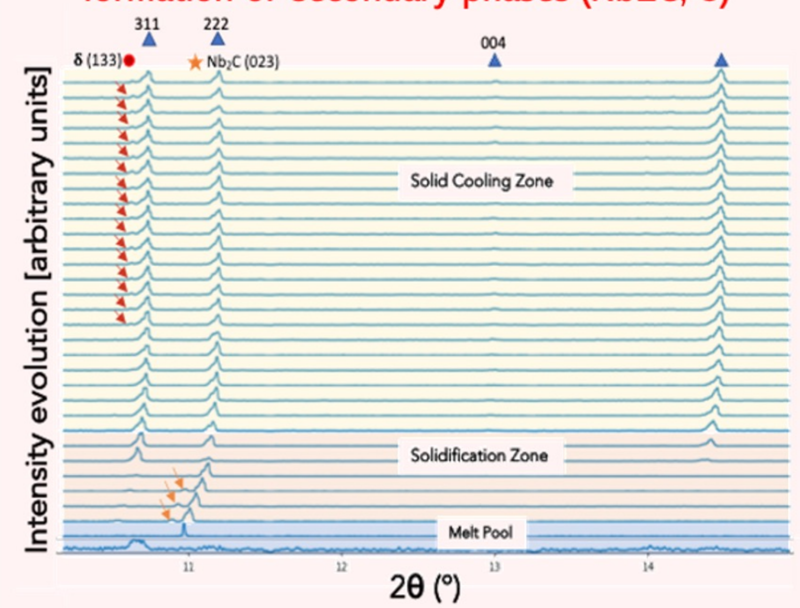




Material: Inconel 625

Energy	61.3 keV
Beam size	250 × 250 μm²
Detector Frame Rate	100 Hz

1D analysis: Inter-dendritic growth and formation of secondary phases (Nb2C, δ)



Verification via post-processing

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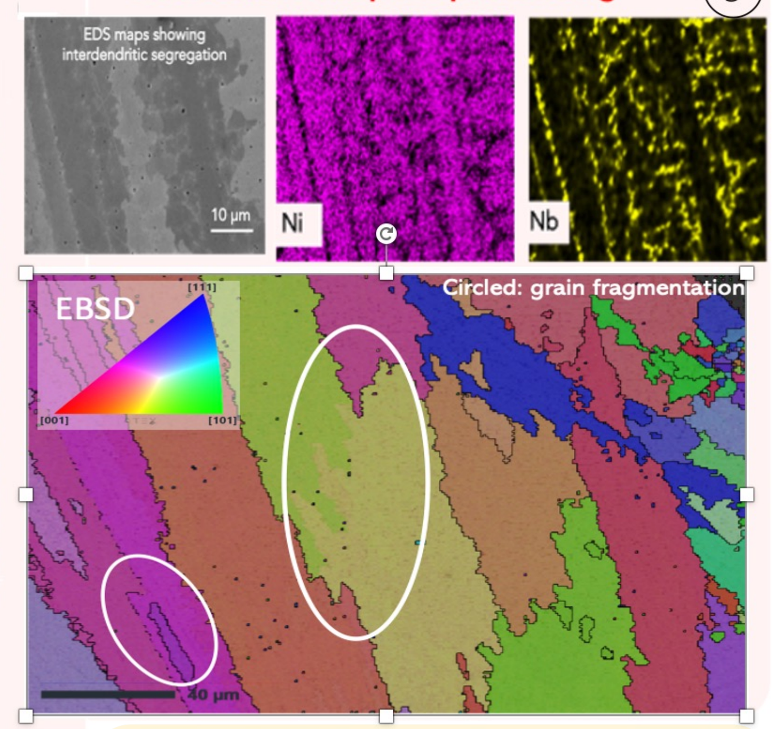
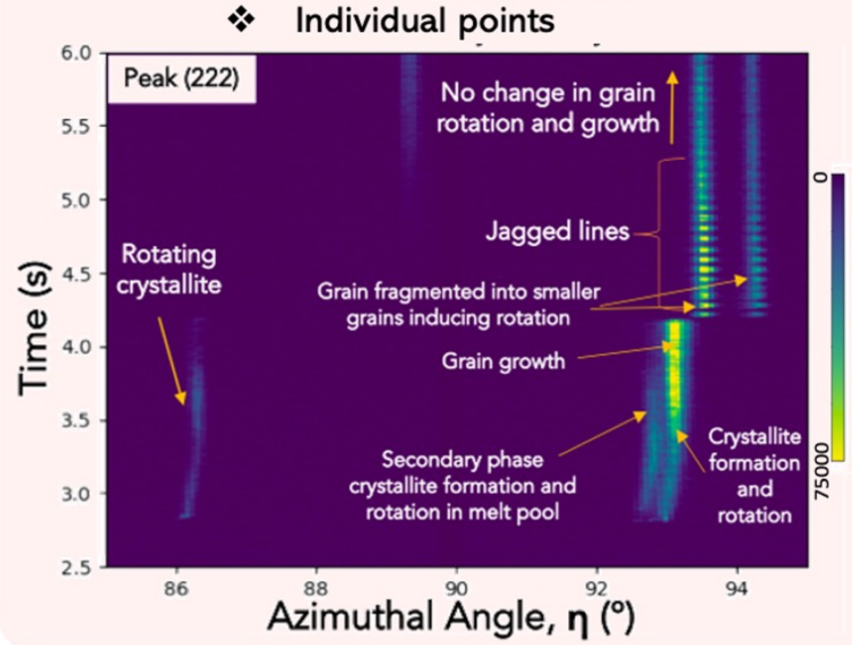


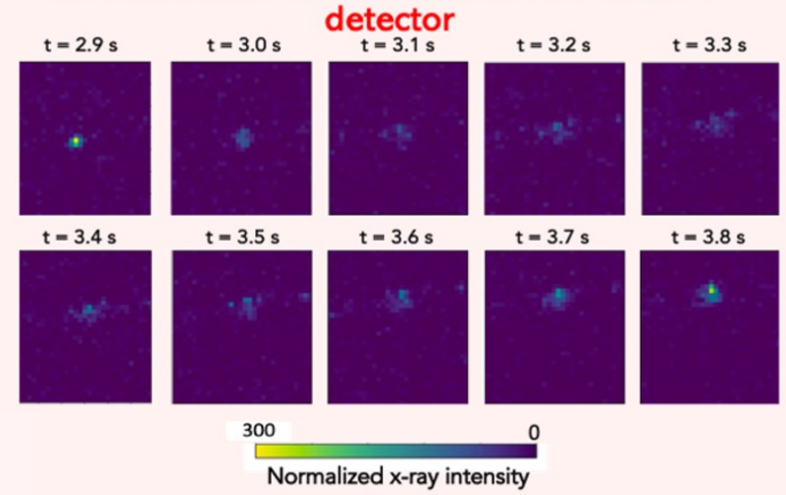
Azimuthal angle (η) versus time plot explaining stages and events during solidification

- ❖ Straight lines (growth)
- ❖ Curved lines (rotation)
- ❖ Individual points

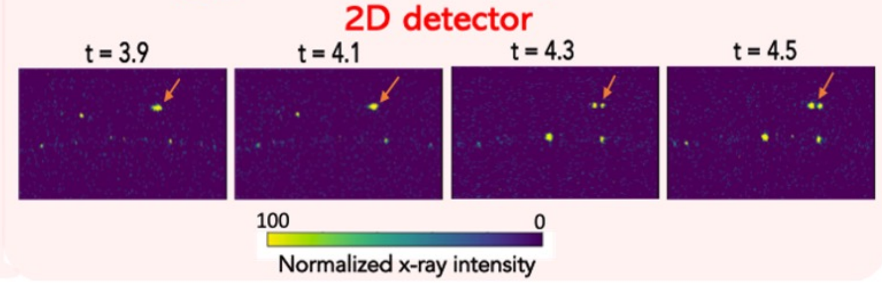


Tracking grain rotation phenomena from 2D detector

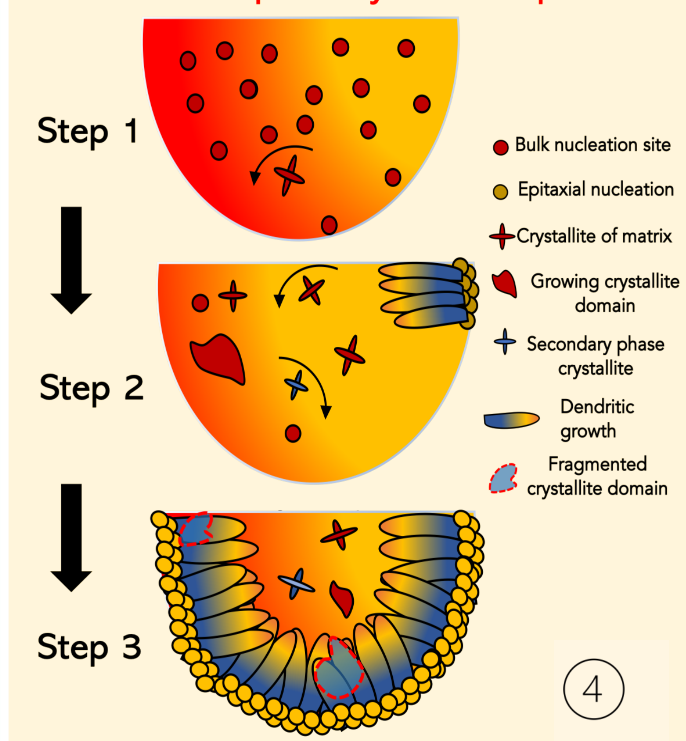
2



Tracking grain fragmentation phenomena from 2D detector



Conclusion: Solidification pathway in AM explained



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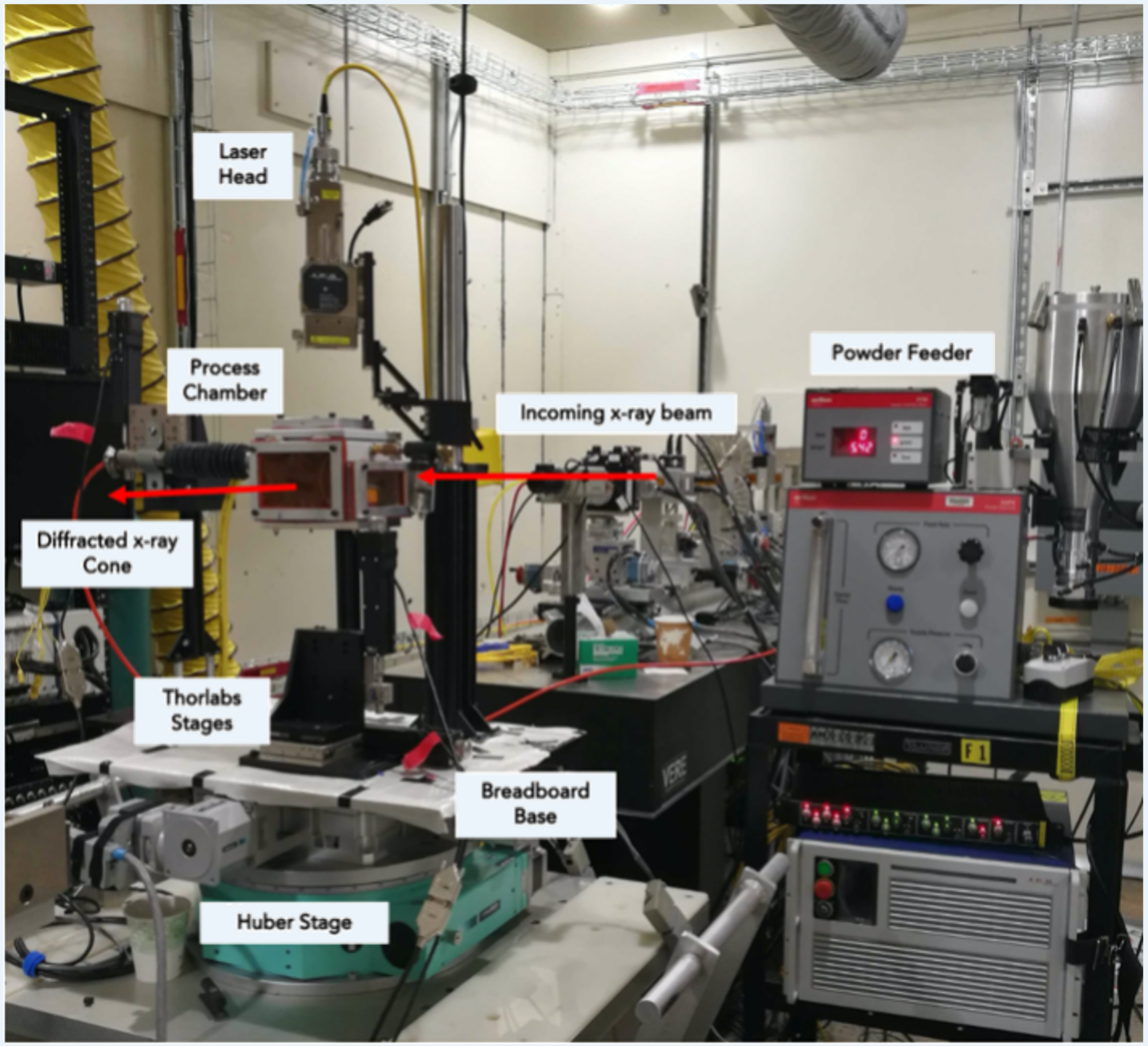
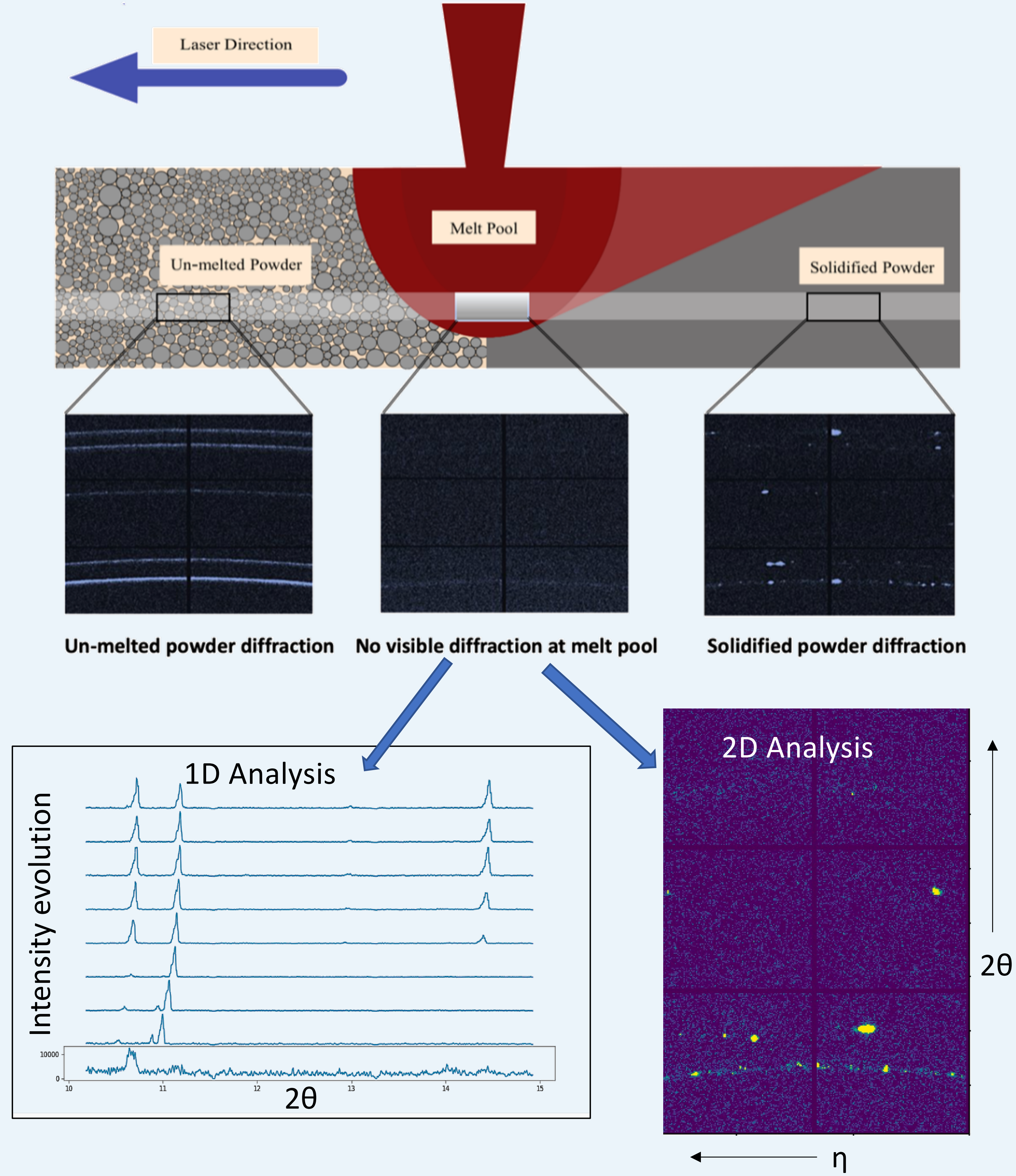
Specialized Equipment for CHESS

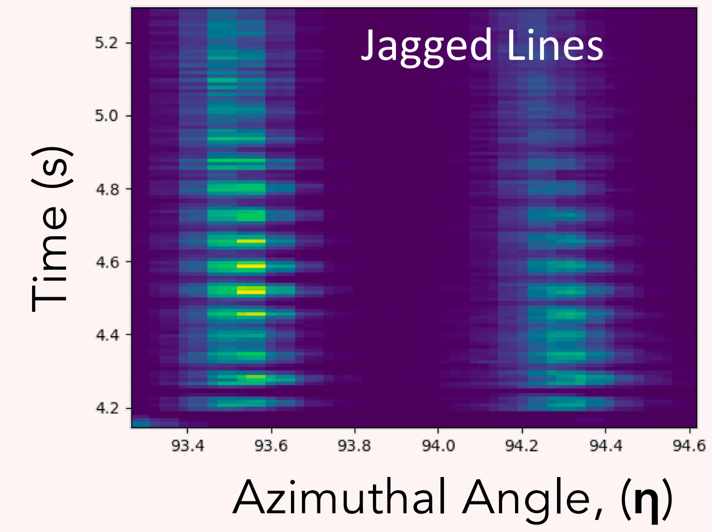
- Enabling studies of the fundamentals of the solidification process during AM

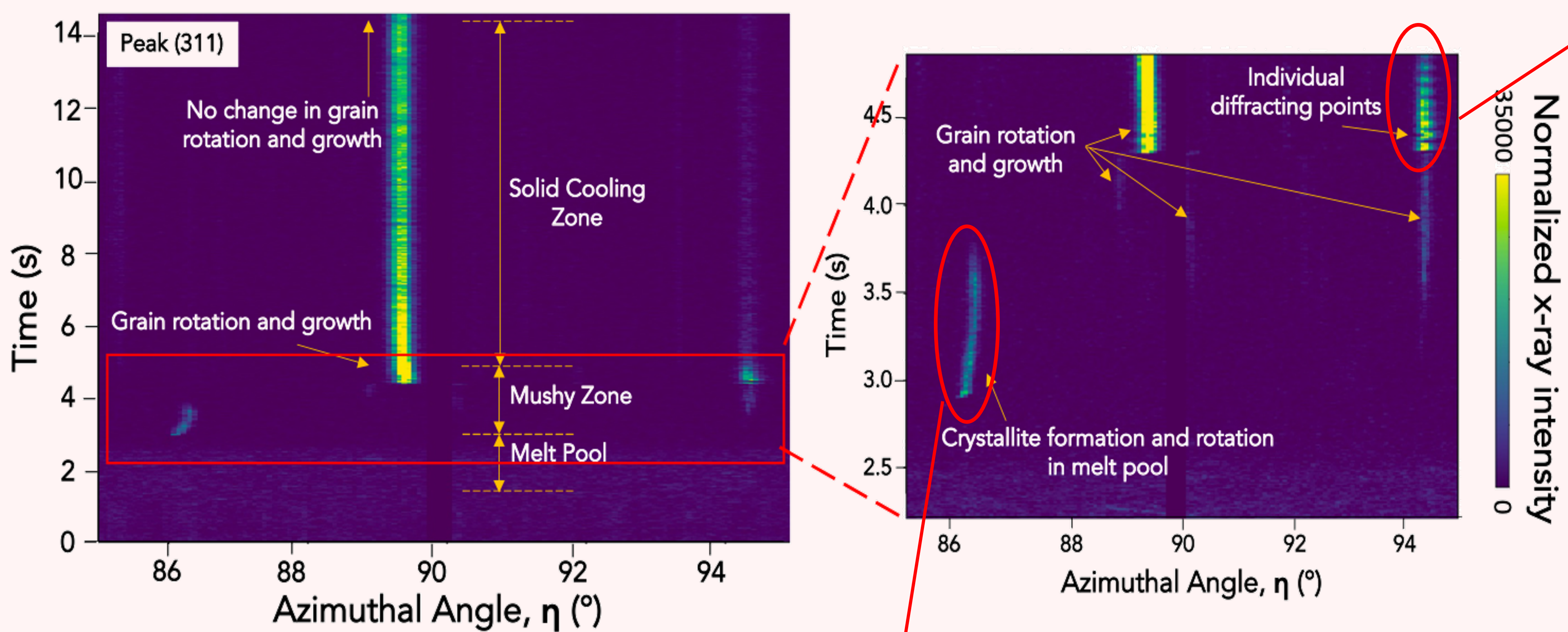




Crystallite formation, growth and rotation, jagged nature lines – Azimuthal plots from 2D detectors

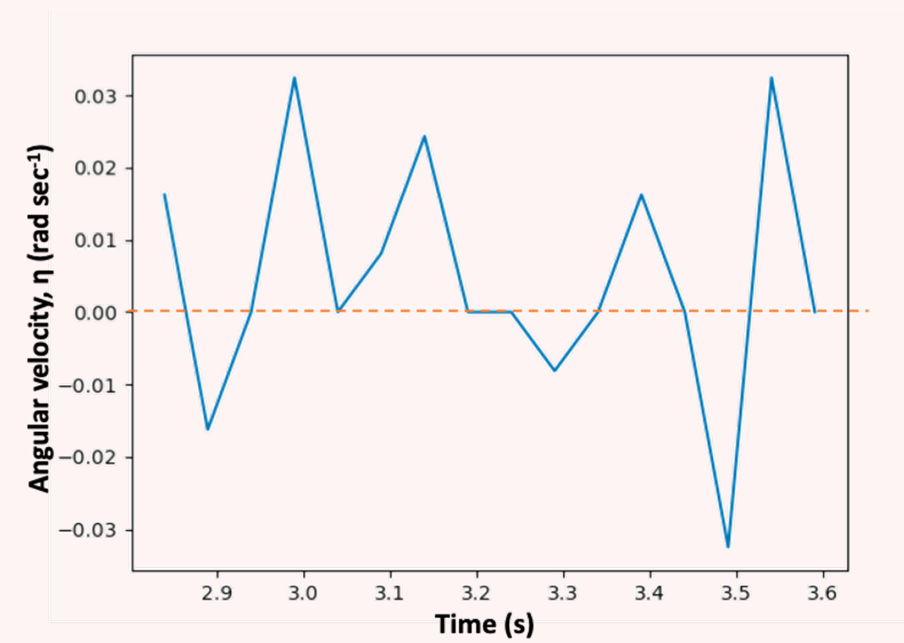
Usually, there are three distinct features as we see from our plots:
1. individual reciprocal lattice points;
2. straight vertical lines (signifying grain growth)
3. curved vertical lines (indicating grain rotation) at a certain angular velocity





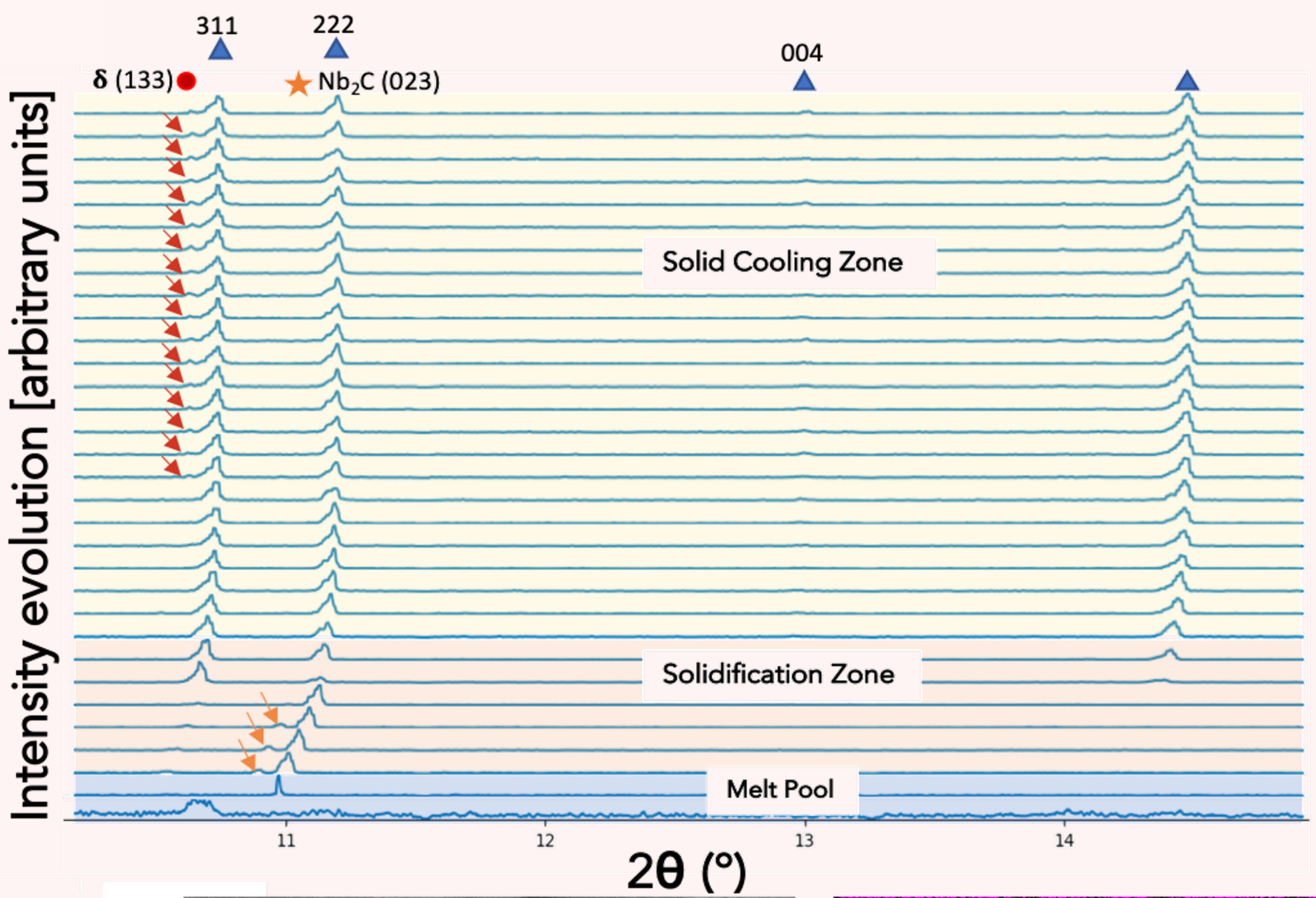
- The frequency range of ~16.7 Hz indicate it could be a periodic of movement of the fluid in the mushy zone, moving the already grown dendrites in and out of the diffraction condition

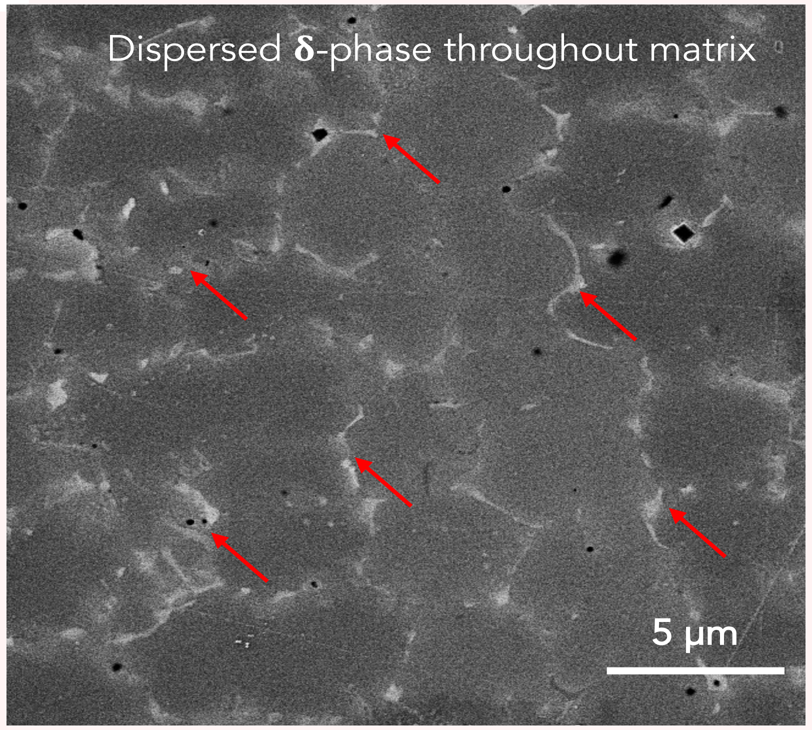
- Movement of the crystallite in the semi-solid region depends on the convection effects in the melt pool
- The change in direction of angular velocity can inform the clockwise or anti-clockwise convection effects

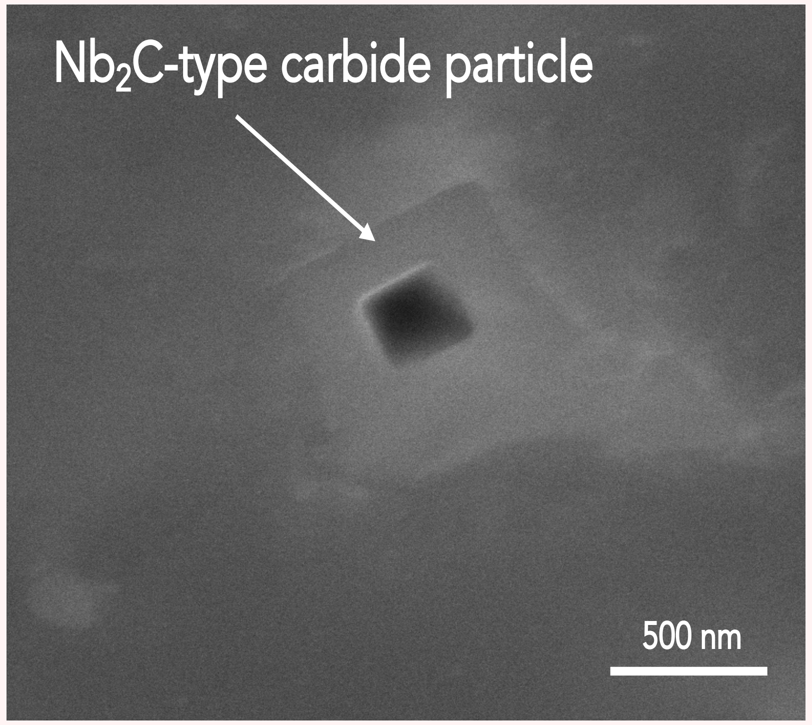


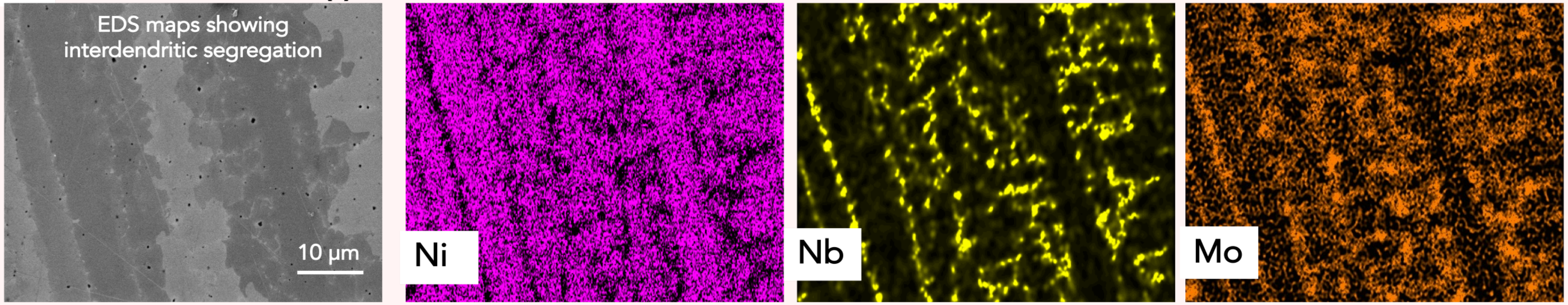
Inter-dendritic growth and formation of secondary phases

- The dendritic + inter-dendritic growth follow a staggered approach
- This accompanied by formation of secondary phases (red and orange arrows) (verified by EDS)
- The asymmetric shoulders of the peaks are due to the inter-dendritic growth, proven by EDS



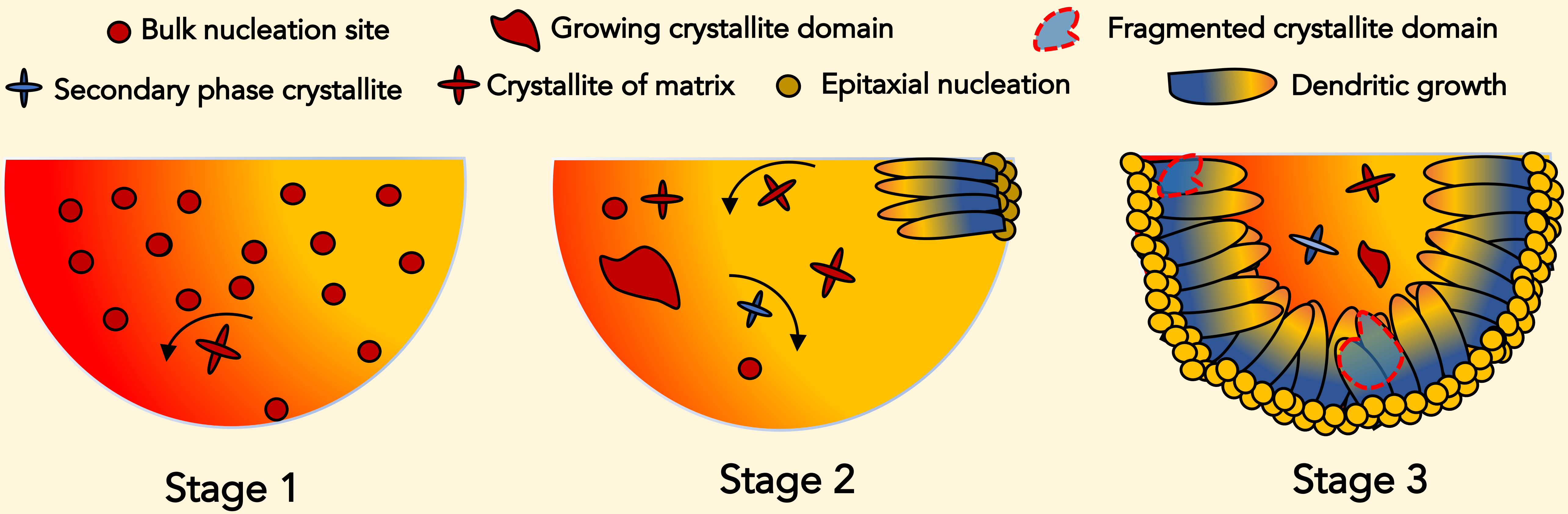






Mechanisms of grain development – Nucleation and formation, rotation, dendritic growth

Mechanism: bulk nucleation, crystallite formation and rotation and dendritic growth and formation of secondary phases



- The first step is
  - ✓ Undercooling induced heterogeneous nucleation in the melt pool
  - ✓ Formation of nuclei (stray dendrites) act as catalyst to grain formation
- The second step is
  - ✓ Crystallites may rotate with a certain velocity in the melt pool liquid, grow or break into smaller fragments
  - ✓ the growth of the smaller grains into larger grains, from stray equiaxed dendrites/crystallites in the melt pool to grow as dendritic channels.
- The third step is
  - ✓ followed by the growth of the inter-dendritic region, which solidifies the remaining inter-dendritic network