CANFSA

Center for Advanced Non-Ferrous Structural Alloys
An Industry/University Cooperative Research Center

Spatially Resolved Acoustic Spectroscopy (SRAS)

Spring 2019 Semi-Annual Meeting
Iowa State University, Ames, IA
April 3-5, 2019

Principal Developer: Thomas K. Ales (ISU)
Postdoc: Maria J. Quintana (ISU)
Faculty: Peter C. Collins (ISU)
Other Participants: Lucas Koester (ISU), Dan Barnard (ISU)
SRAS Inventors and Experts (U. Nottingham): Matt Clark, Richard Smith and Wenqi Li

Funded under ONR DURIP project N00014-17-1-2294 "Analytical serial sectioning with orientation microscopy capabilities"

Spatially Resolved Acoustic Spectroscopy (SRAS)

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- Principal Developer: Thomas Ales (ISU)
- Postdoc: Maria Quintana (ISU)
- Advisor(s): Prof. Peter Collins (ISU)

- Problem: Need for a new instrument that can rapidly analyze crystal orientations at the 'mesoscale' (beyond the nanometer and micrometer length scales).
- Objective: Establish and demonstrate a new 3D characterization tool to integrate microstructure, texture and defects, involving the use of surface acoustic waves.
- Benefit: Instrument that measures crystal orientations at the 'mesoscale' and eventually in 3D datasets.

Project Duration

August 2017 to June 2020

Recent Progress

- The optic train is assembled and its alignment is in progress
- Forward model code is almost finished
- Transfer system from Robomet to SRAS is designed

Metrics		
Description	**% Complete**	**Status**
1. Design strategy	100%	●
2. Review of Materials State Awareness	100%	●
3. Mathematics that underpin the forward and inverse models and Coding	95%	●
4. Assembly, alignment and coupling to Robo.met	50%	●
5. 2D and 3D datasets	0%	●

Spatially Resolved Acoustic Spectroscopy (SRAS)

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- Need for a new instrument that can rapidly analyze crystal orientations at the 'mesoscale'
  - Beyond the nanometer and micrometer length scales
- Spatially Resolved Acoustic Spectroscopy (SRAS) has been developed at the University of Nottingham
  - One possibility would involve the use of surface acoustic waves.

OPTICS & PHOTONICS UNIVERSITY OF NOTTINGHAM

University of Nottingham UK | CHINA | MALAYSIA

Spatially resolved acoustic spectroscopy (SRAS): texture and microstructure characterisation

Wenqi Li, Rikesh Patel, Richard Smith, Matthias Hirsch, Matt Clark, Adam Clare and Steve Sharples

Optics & Photonics Group, Faculty of Engineering, University of Nottingham, UK.

QNDE Workshop 2017, Provo, Utah, US

Spatially Resolved Acoustic Spectroscopy (SRAS)

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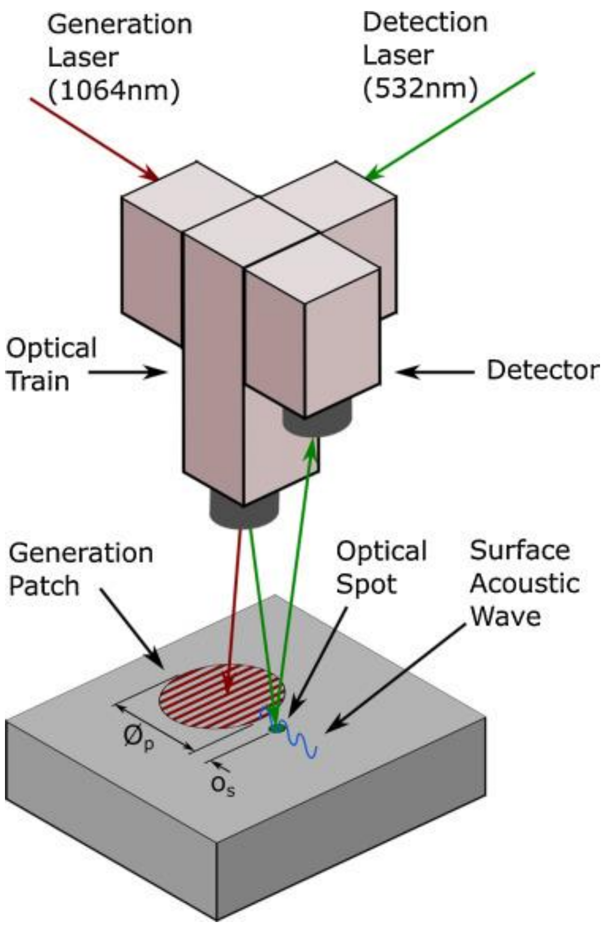


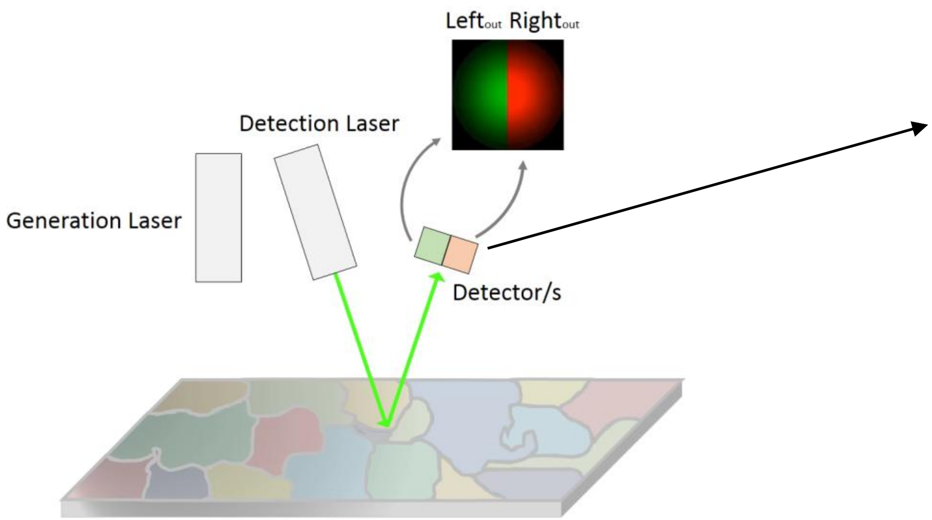
- Laser based microscopy to obtain texture information for a large sample
- A pulsed laser is imaged to the sample surface, where the absorbed pulses generate acoustic waves that have a characteristic frequency *f*
  - This frequency is measured by monitoring the interaction of the acoustic waves with a probe laser and is then processed to recover the surface wave acoustic velocity (SAW)
  - The SAW velocity varies with grain orientation giving information about the grain size and orientation distribution
- The sample is raster scanned to build up an image
- If multiple velocity images are taken with different acoustic wave propagation directions then it is possible to recover the actual orientation of the grains

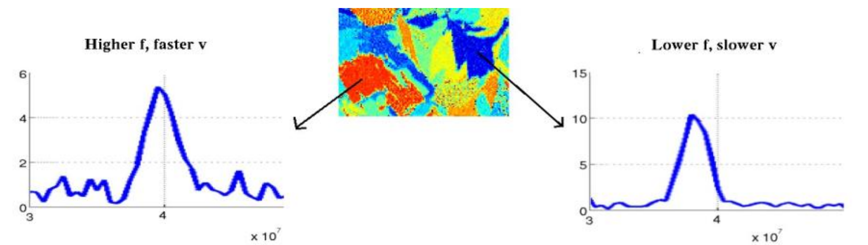
Richard J.S. et al, J of Material Processing Tech., Vol 236, 2016, pg 93

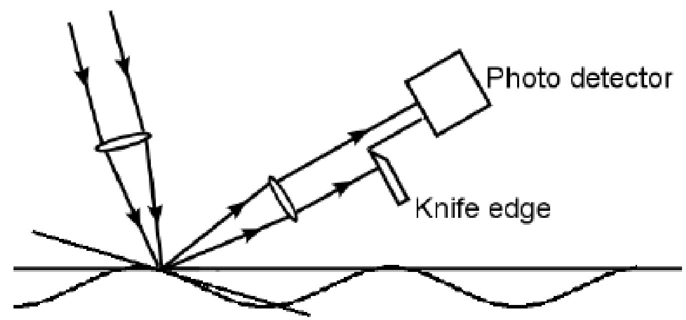
SRAS theory

- The size of the grating patch defines your resolution
- The direction of the lines defines the direction of wave propagation
- v = fλ

Frequency → Velocity





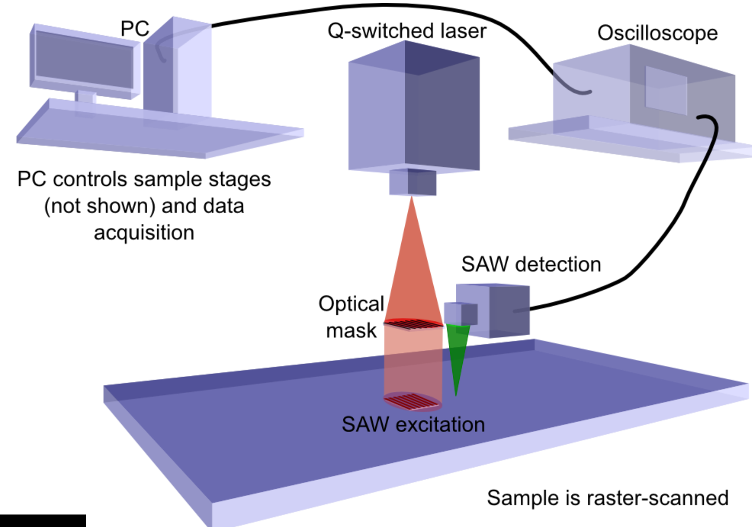


(Patent No.: GB2441953, 2009)

SRAS instrument

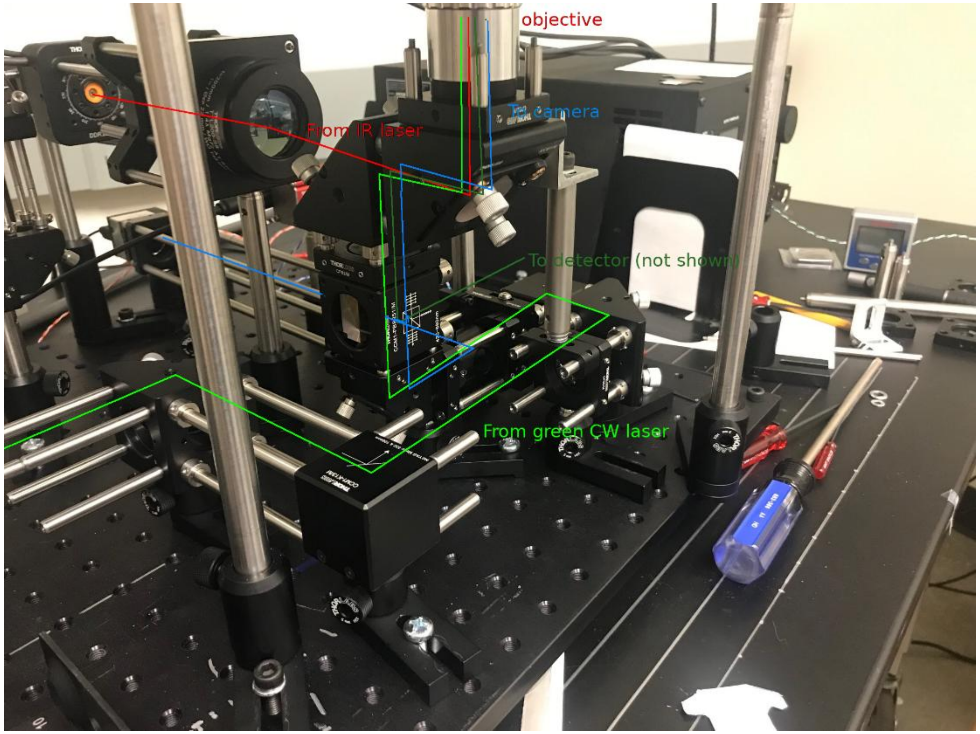
- At each point generate SAWs using laser and a grating – fixed acoustic wavelength
- Detect the SAWs with another laser
- Find the peak of the frequency spectrum of the detected waves

[redacted]



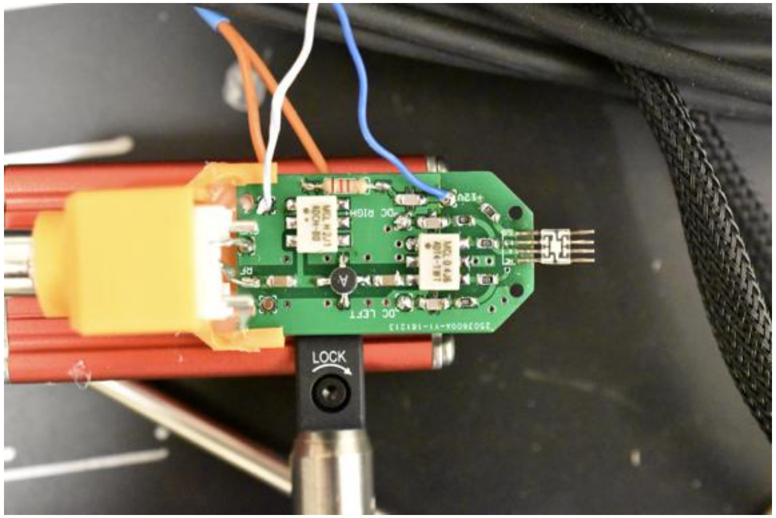
- Calculate the velocity using v = fλ

SRAS instrument



Current state of optical train

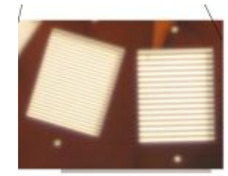
Custom made printed circuit boards

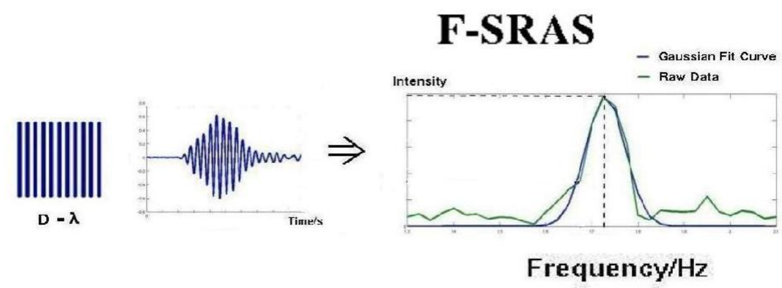


Center Proprietary – Terms of CANFSA Membership Agreement Apply

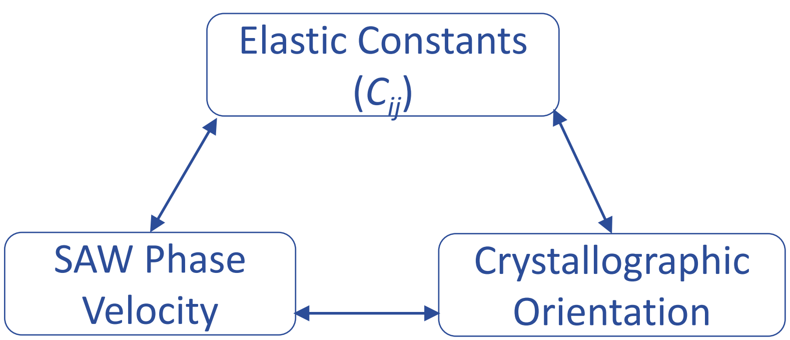
Orientation Determination

- $v = f\lambda$
  - λ is the fringe spacing of the mask
    - Mask is glass coated with Cr
    - Grating acts as a filter.
  - f is the peak frequency from the experiment
    - Velocity measured
    - FFT, interpret frequency domain to determine peak





Elastic constants, SAW Phase Velocity, and Crystallographic Orientation are interrelated.

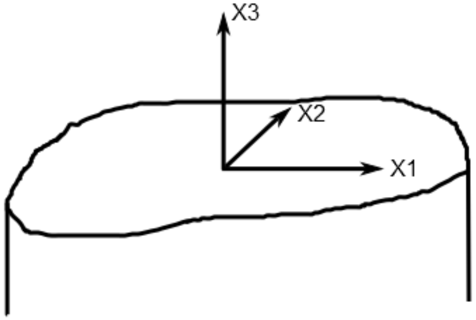


- **EASY:**
  - Calculating the elastic constants
  - Predicting SAW Phase Velocity
- **HARD:**
  - Calculating crystallographic orientation
- **SOLUTION:**
  - Predict SAW velocity, index against solutions

Predict SAW velocities

- Wave equation:

$$\rho \frac{\partial^2 u_j}{\partial t^2} = \frac{\partial T_{ij}}{\partial x_i},\ (i, j = 1, 2, 3)$$



- Boundary condition:

$$T_{3j} = c_{3jkl}\partial u_k/\partial x_l = 0,\ \text{for } j = 1, 2, 3.$$

- Solution of the wave equation:

$$u_i = \sum_{n=1}^{3} C_n \alpha_i^{(n)} \exp[-j\beta l_3^{(n)} x_3] \exp[j(\omega t - \beta l_1 x_1 - \beta l_2 x_2)]$$

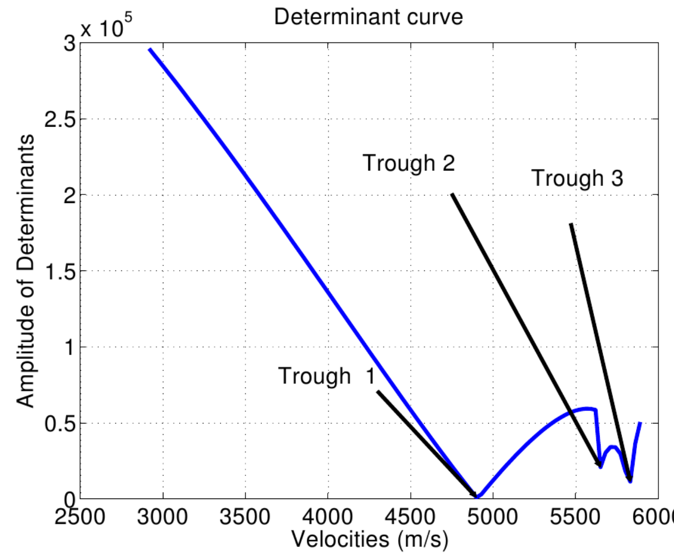
- Determinant of the boundary condition:

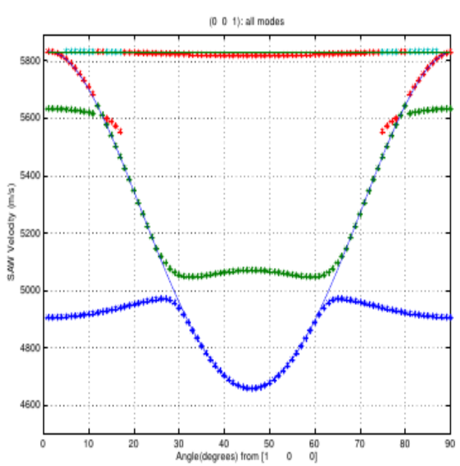
$$d_{mn} = c_{m3kl}\alpha_k^{(n)} l_l^{(n)} = 0,\ \text{with } l_1^{(n)} \equiv l_1,\ l_2^{(n)} \equiv l_2.$$

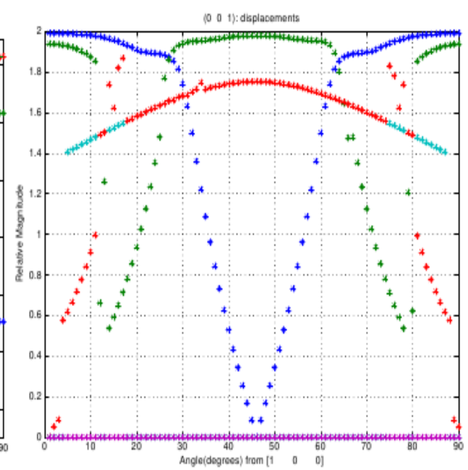
- Li, W., Thesis, Laser ultrasonic method for determination of crystallographic orientation of large grain metals by spatially resolved acoustic spectroscopy (SRAS), University of Nottingham, 2012
- Farnell, G.W., Properties of Elastic Surface Waves, *Physical Acoustics, 6* (1970): 109-166
- Viktorov, I.A., Rayleigh and Lamb Waves: Physical Theory and Applications, Trans. From Russian. Plenum Press, 1967

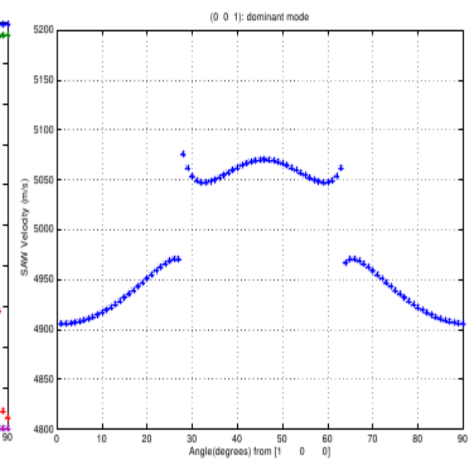
Predict SAW Velocities

- The determinant of the boundary condition is calculated at a specific velocity
- The curve of the determinant is plotted as a function of the velocity
- Troughs of the curve correspond to the wave modes
- The ‘dominant’ mode is selected according to the out-of-plane displacement









Forward Model

- Root Finding
  - Involves solving for *v* for the selected microstructural anisotropy
  - Calculation of the amplitudes and down selection of the dominant modes of propagation as a function of direction

$$\begin{vmatrix} \Gamma_{11} - \rho v^2 & \Gamma_{12} & \Gamma_{13} \\ \Gamma_{21} & \Gamma_{22} - \rho v^2 & \Gamma_{23} \\ \Gamma_{31} & \Gamma_{32} & \Gamma_{33} - \rho v^2 \end{vmatrix} = 0$$

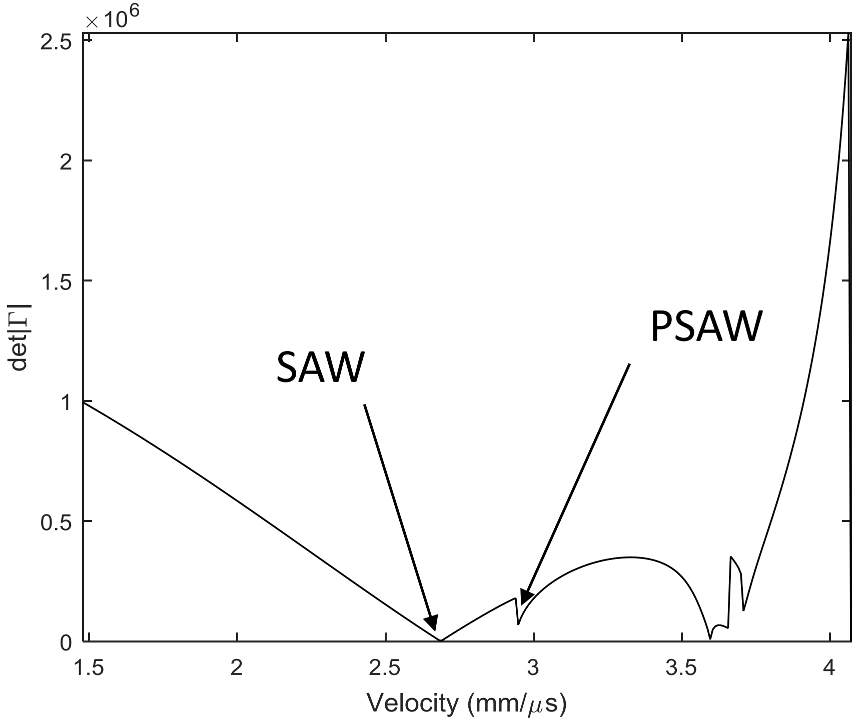
$$\Gamma_{11} = c_{11} + c_{44} l_3^2,$$

$$\Gamma_{22} = c_{44}(1 + l_3^2),$$

$$\Gamma_{33} = c_{44} + c_{11} l_3^2,$$

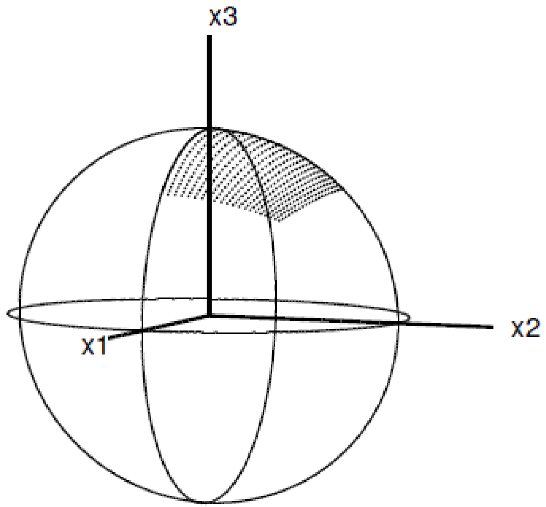
$$\Gamma_{13} = \Gamma_{31} = (c_{11} - c_{44}) l_3,$$

$$\Gamma_{12} = \Gamma_{21} = \Gamma_{23} = \Gamma_{32} = 0.$$

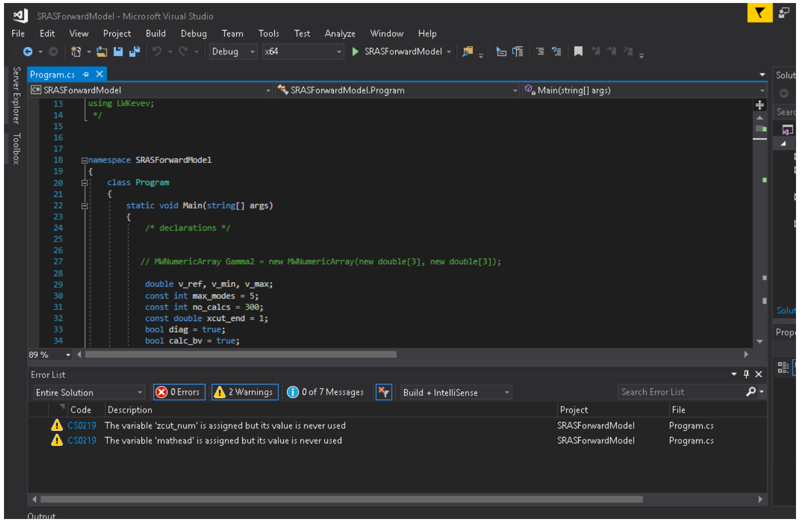


Forward model

- Utilizes crystal symmetry to reduce calculations necessary
- Data is stored and can be calculated in advance for a particular material system

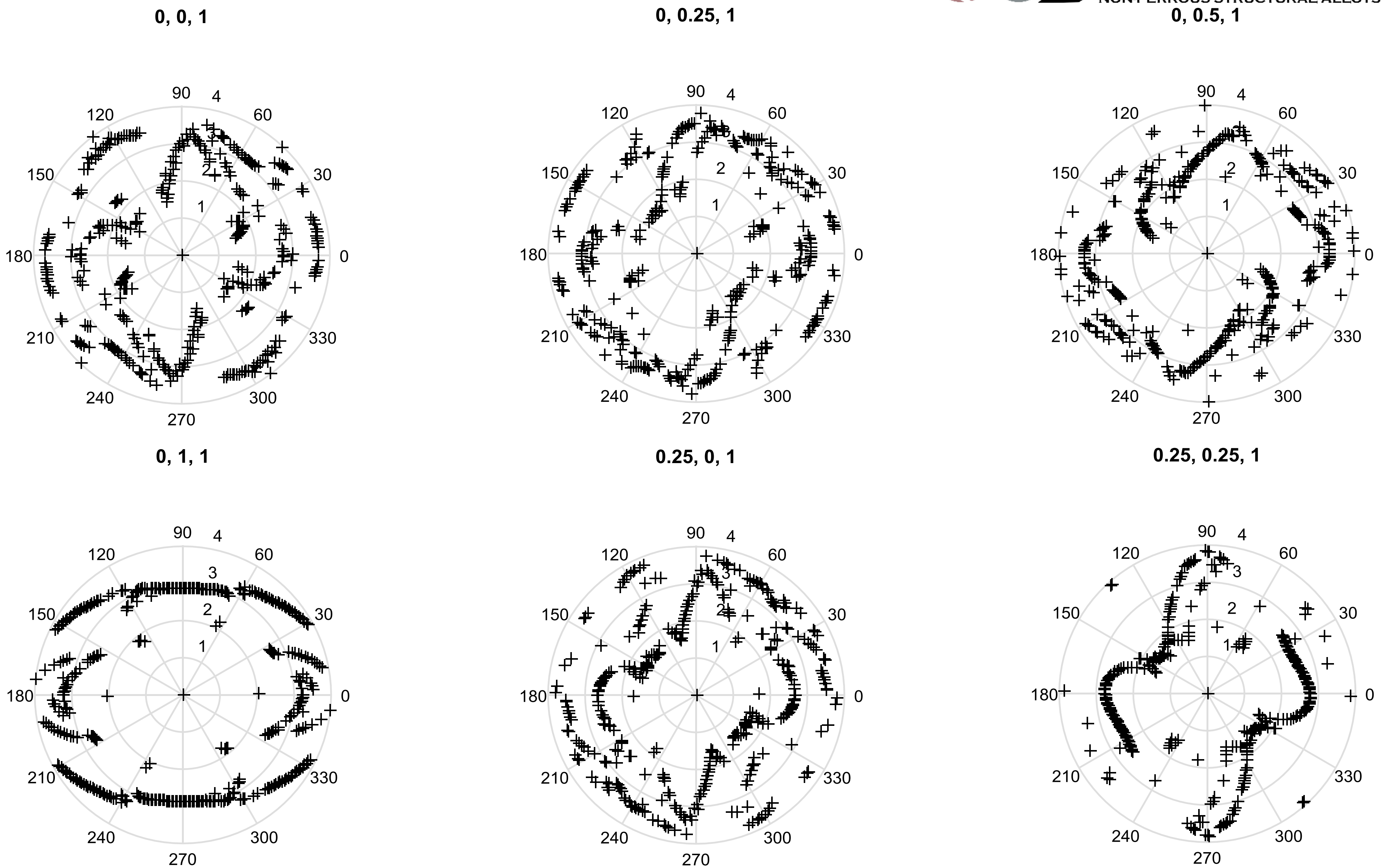


Poles on the unit sphere at which wavespeeds and amplitudes are calculated. All other points are repetitions of these values (cubic symmetry)



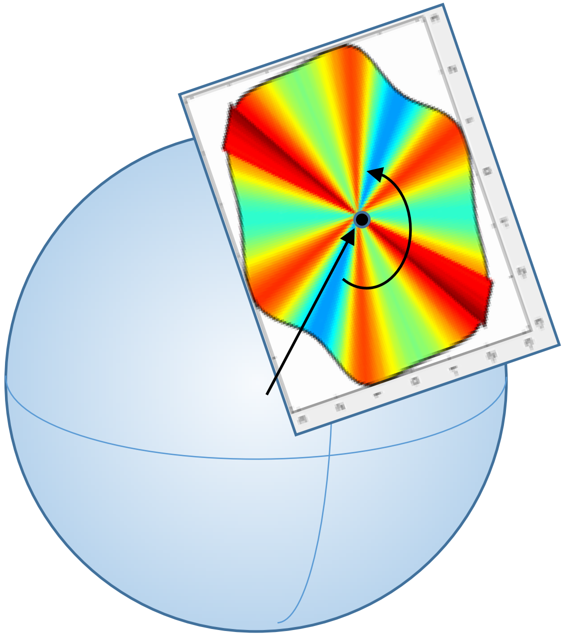
Written in C# for speed and portability

Example of forward model in Nickel

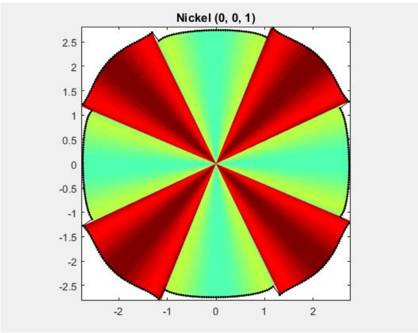


SRAS Forward Model Result

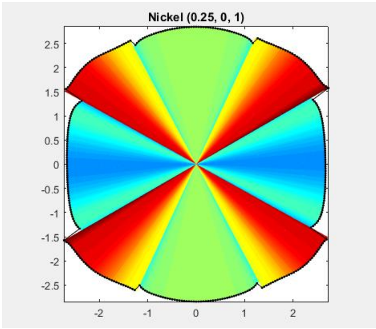
- Example at a single crystal plane shown at right
- All directions (0:180 + symmetry)
- Speeds of dominant modes saved for fitting
- Calculated at 1° intervals for 120 planes



(0,0,1)

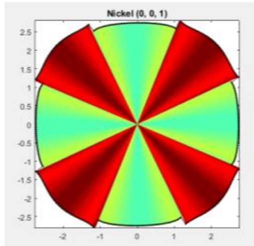


(0.25,0,1)

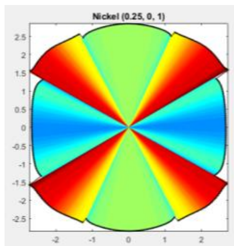


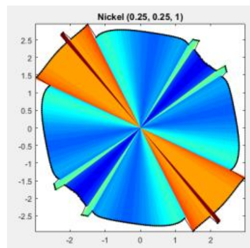
Example of forward model in Nickel

(0,0,1)

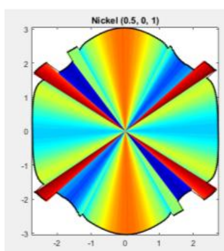


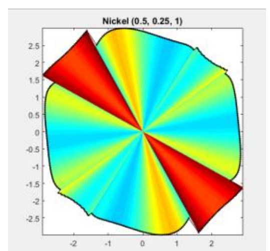
(0.25,X,1))

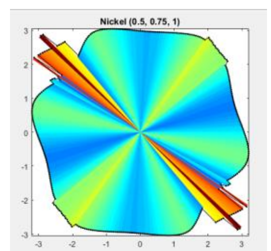




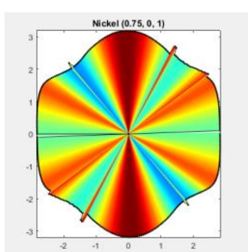
(0.5,X,1))

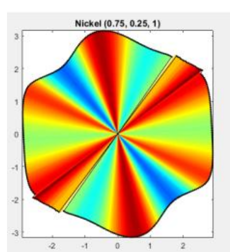


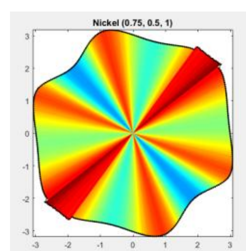


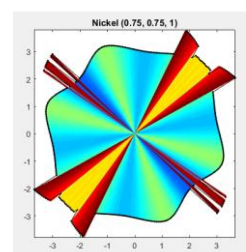


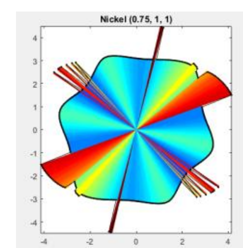
(0.75,X,1))



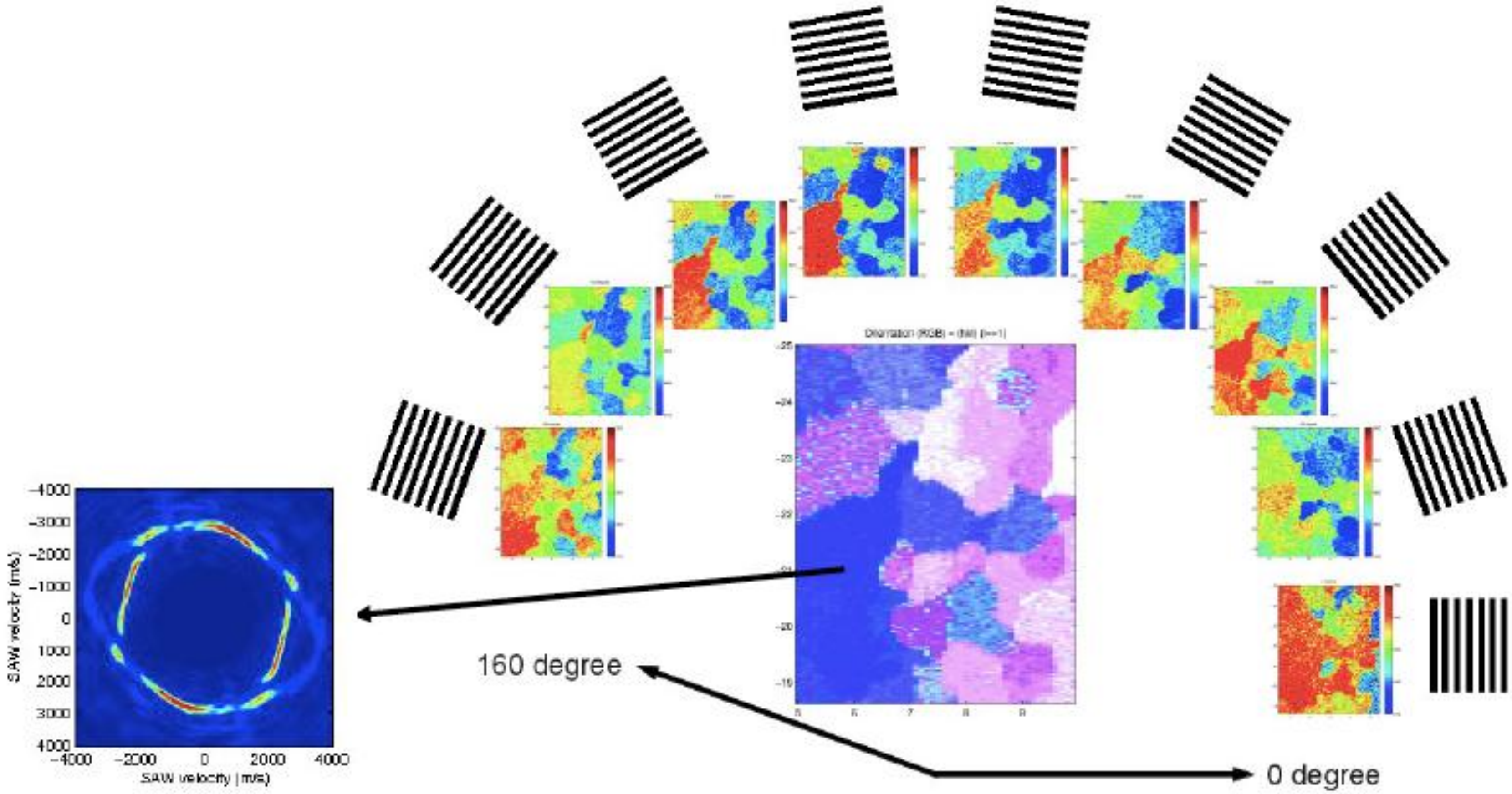






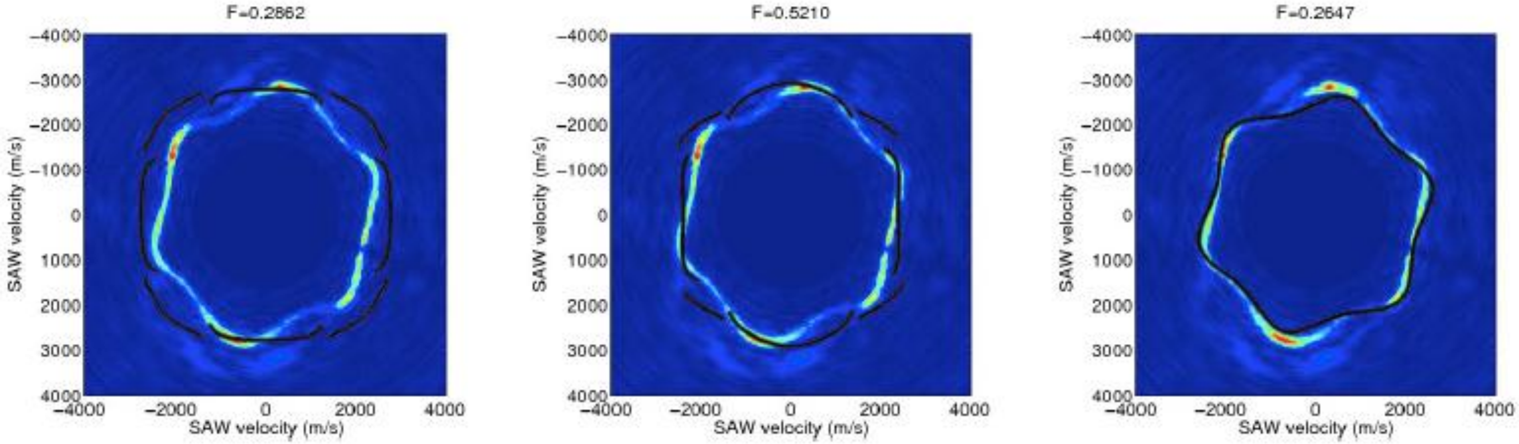


Collect data



Orientation imaging and fit to data

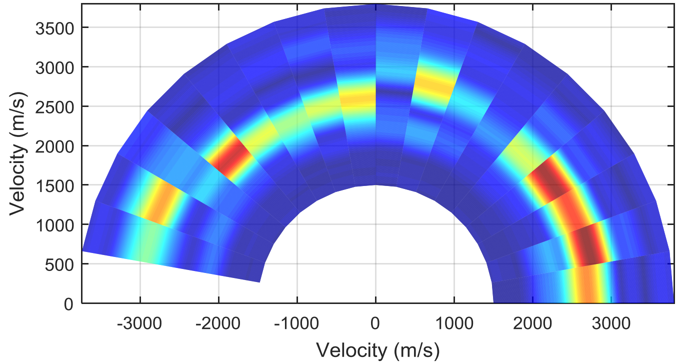
$$F(h,k,l,\theta) = \sum_{\phi=0}^{\phi=\frac{n-1}{n}\pi} A(\phi, v_{(h,k,l,(\phi-\theta))}),\ (n = 1,\ 2,\ 3,\ \ldots)$$

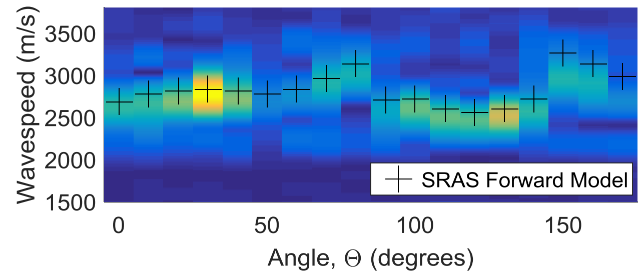


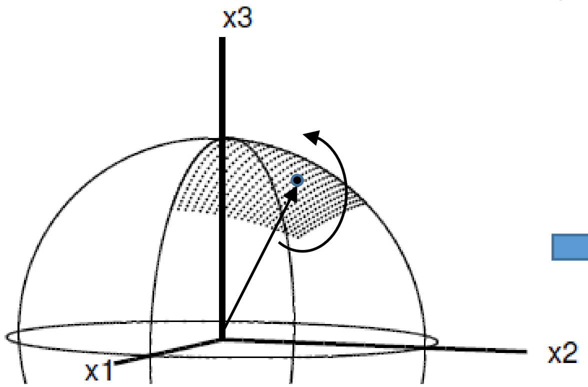
- The merit function is simply the sum of the amplitude under the black '+' on the graphs
- This procedure is repeated for all combinations of plane and propagation direction

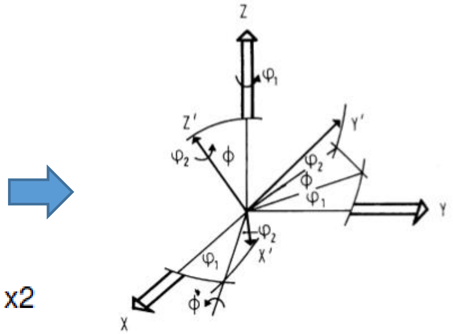
Forward Model Inversion

- Raw data consists of wavespeeds and amplitudes vs direction
- Model data is interpolated onto experimental data and a best fit is found
- Fit includes crystal plane + rotation and can be converted to Euler angles, IPF map, etc. as seen in EBSD data

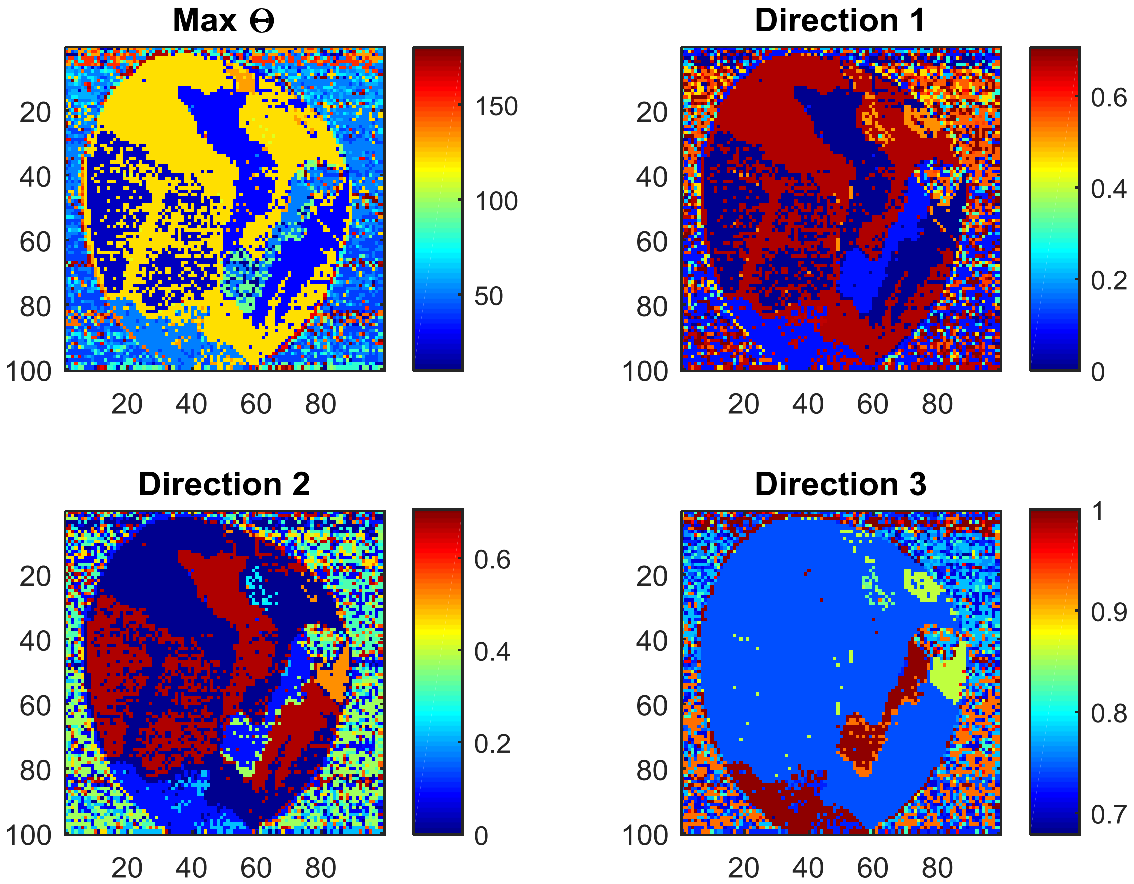






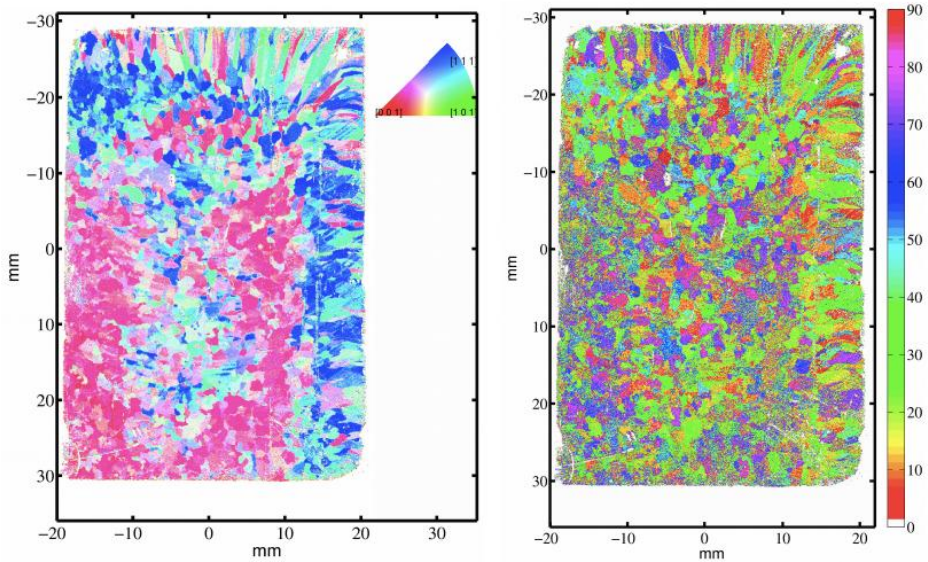


Example of Forward Model Inversion on Nickel

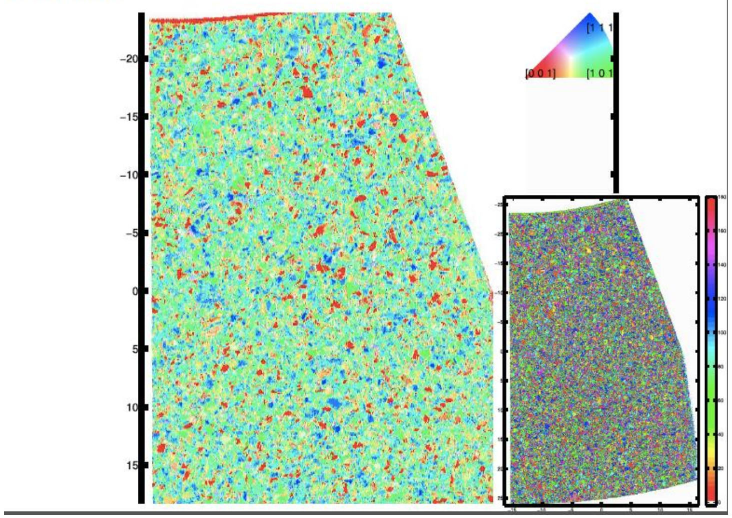


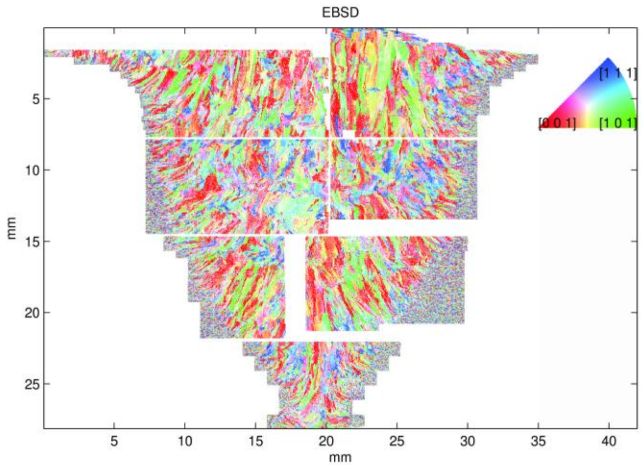
Examples – U. Nottingham

Large grain aluminium



Inconel





Stainless Steel

Hexagonal materials: SRAS on Ti 64

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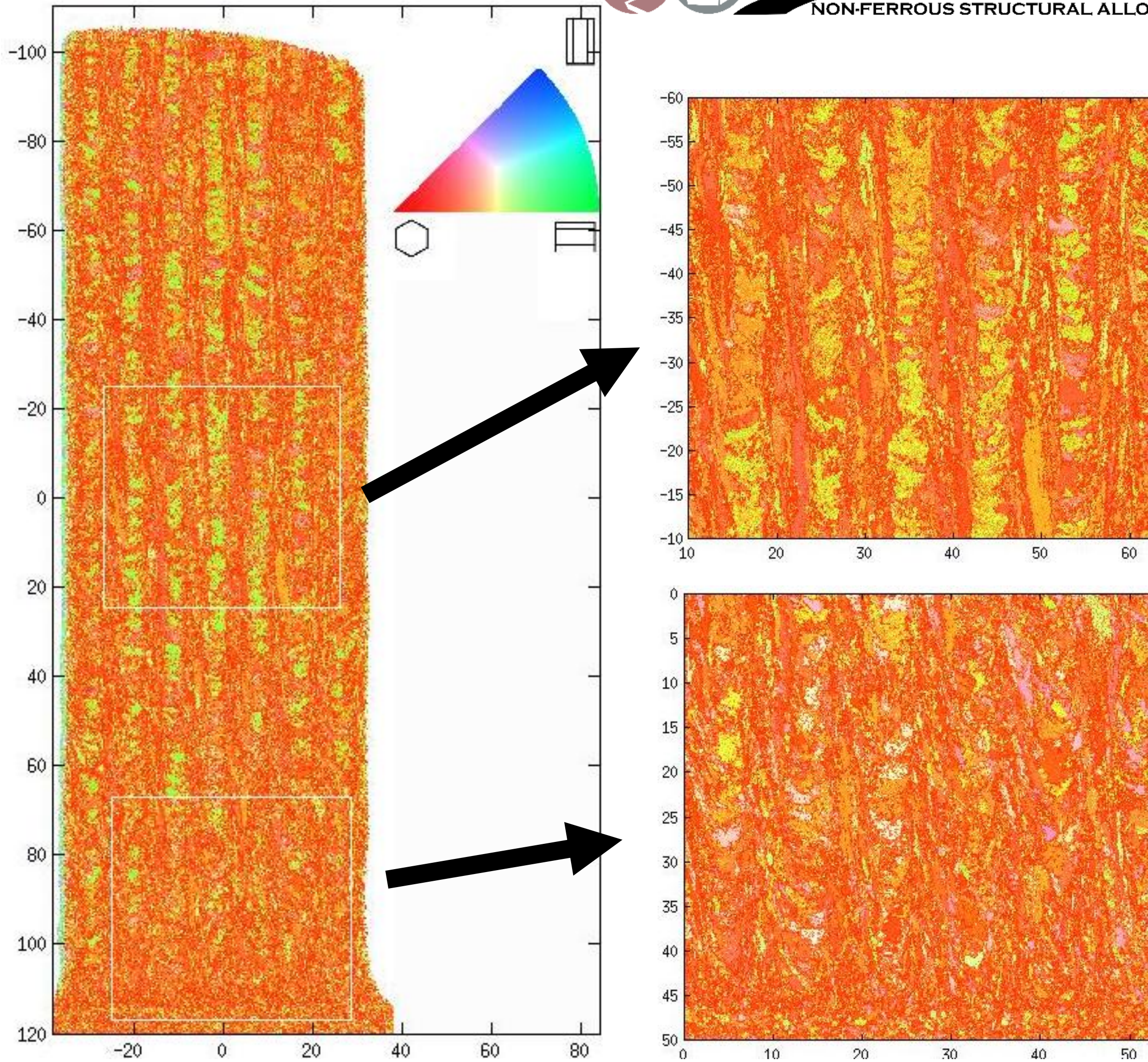
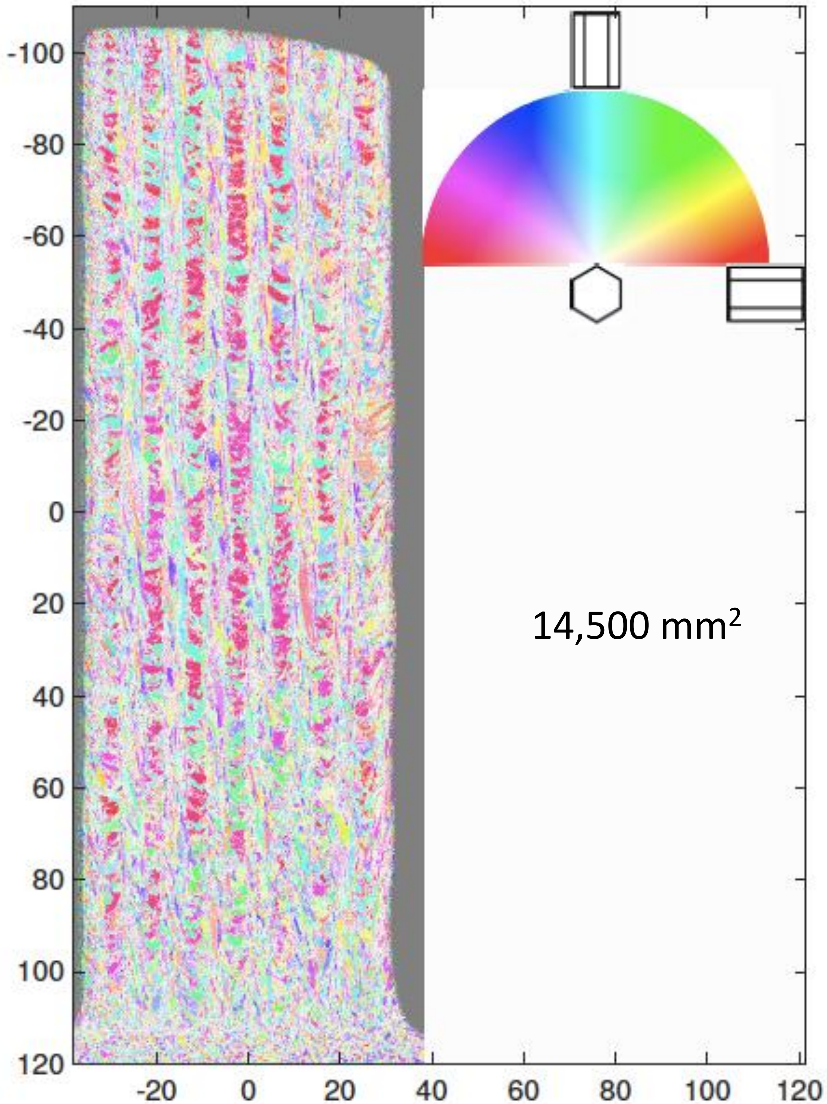
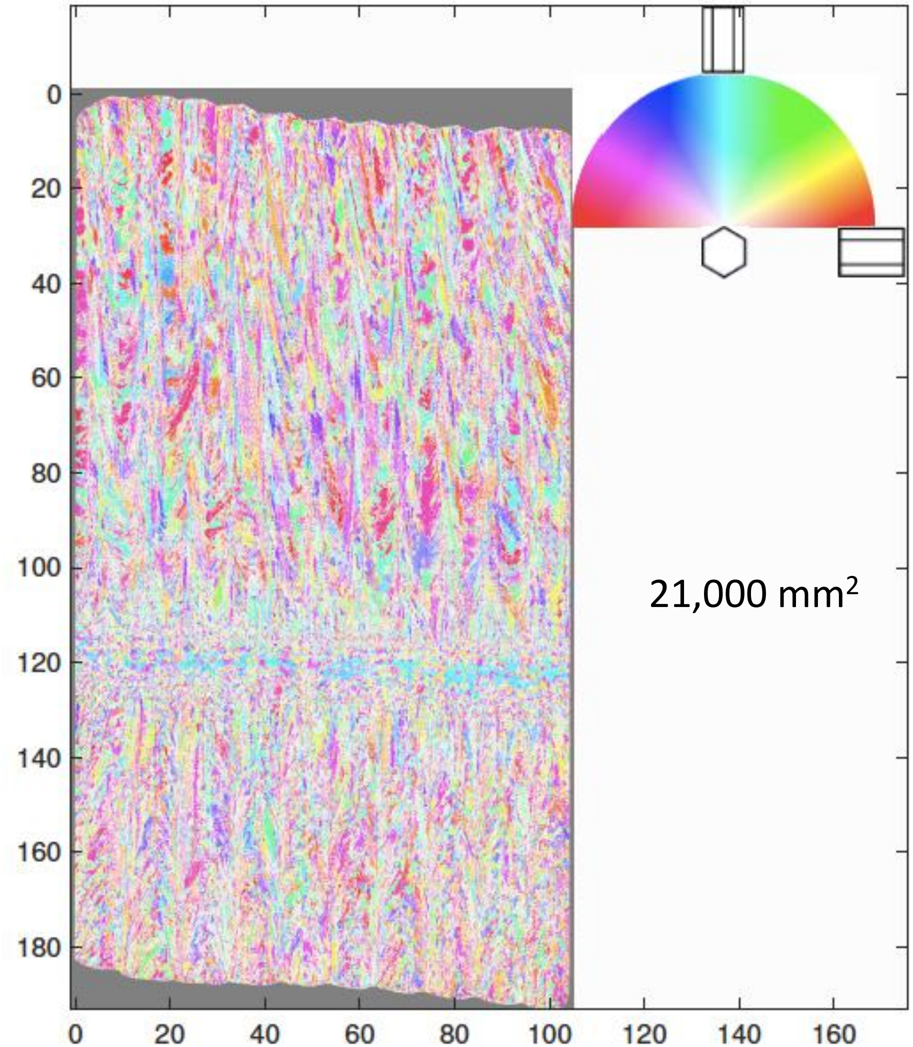
- ~14,500 mm^2
- This scan was obtained in 16 hours.
- A conventional SEM with EBSD, operating under “normal” modes, would take ~10 years of 24 hour operation to obtain this scan.



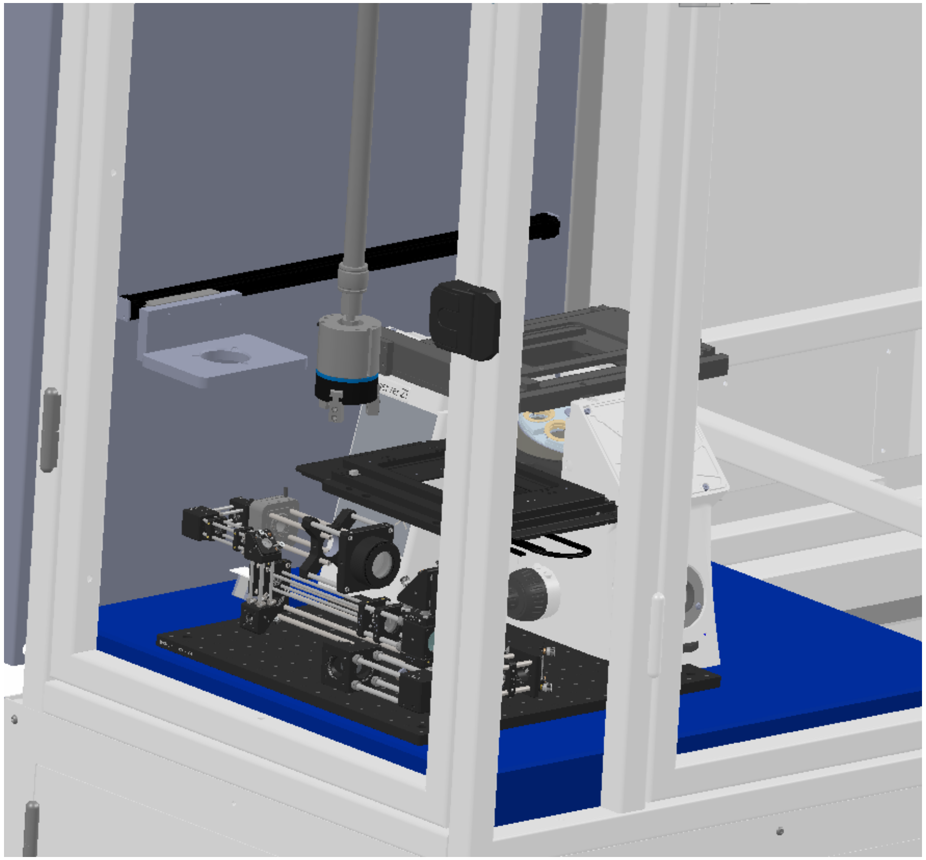
Hexagonal materials: SRAS on Ti 64





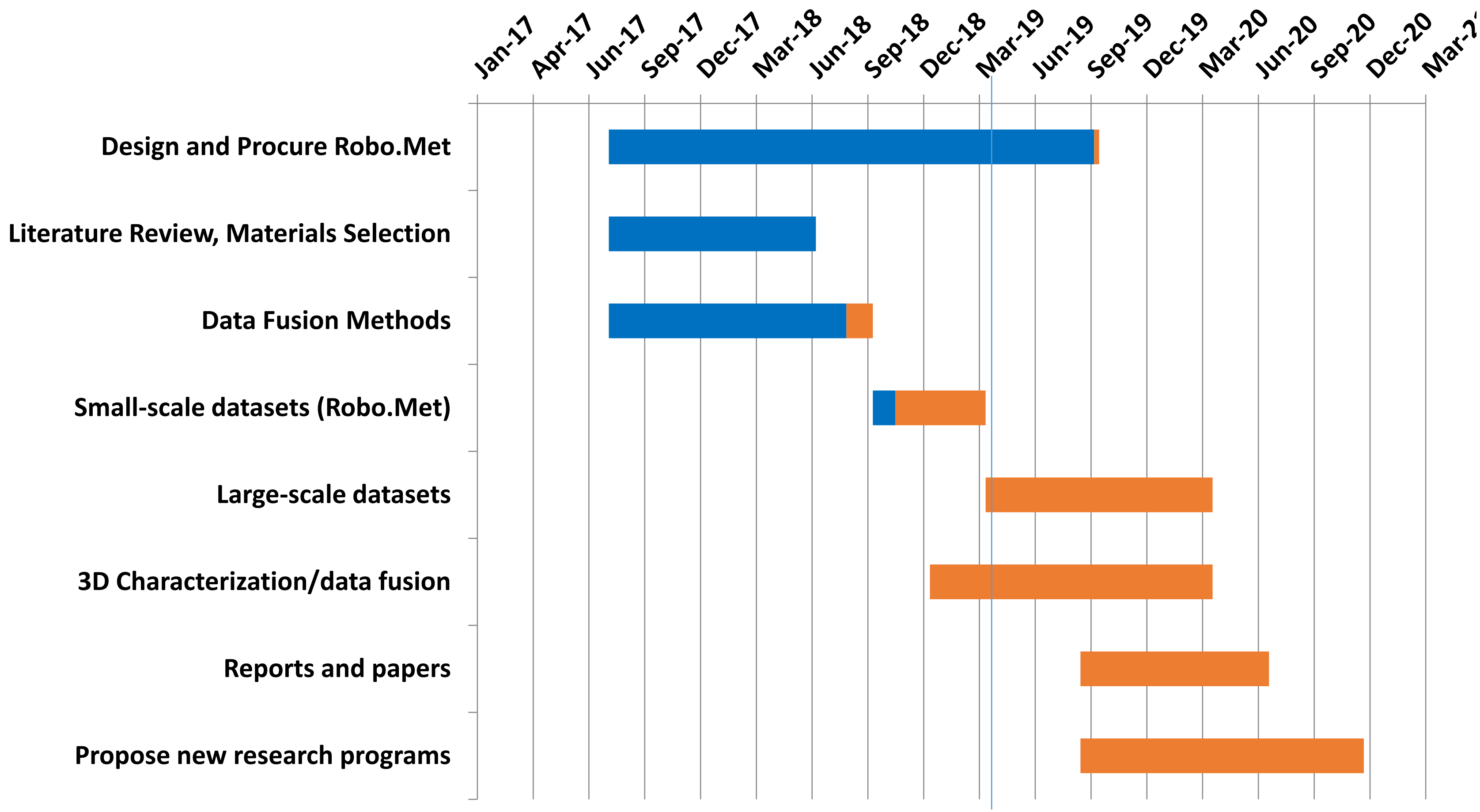
Integrating SRAS onto a Robo.Met-3D platform

Integrating SRAS onto a Robo.Met-3D platform



- Evolution of plans:
  - Integrate the laser into the optical microscope (Zeiss wouldn't enable it)
  - Build a SRAS system to fit in the Robo.Met with a simple pneumatic transfer mechanism (where we are – space is very tight; laser-safe). Need to include z-axis on final optic.
  - Possibly replacing the optical microscope with a SRAS/OM system.

Gantt chart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Design and Procure Robo.Met
Literature Review, Materials Selection
Data Fusion Methods
Small-scale datasets (Robo.Met)
Large-scale datasets
3D Characterization/data fusion
Reports and papers
Propose new research programs

Thank you!

Maria J. Quintana
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