3DXDM

Three Dimensional X-Ray Diffraction Microscopy

My Goal: develop a simulation and control system for a technique to map polycrystalline grain microstructure non-destructively in 3D.

Overall Goal: Integrate this into an actual experimental setup for automated data-taking

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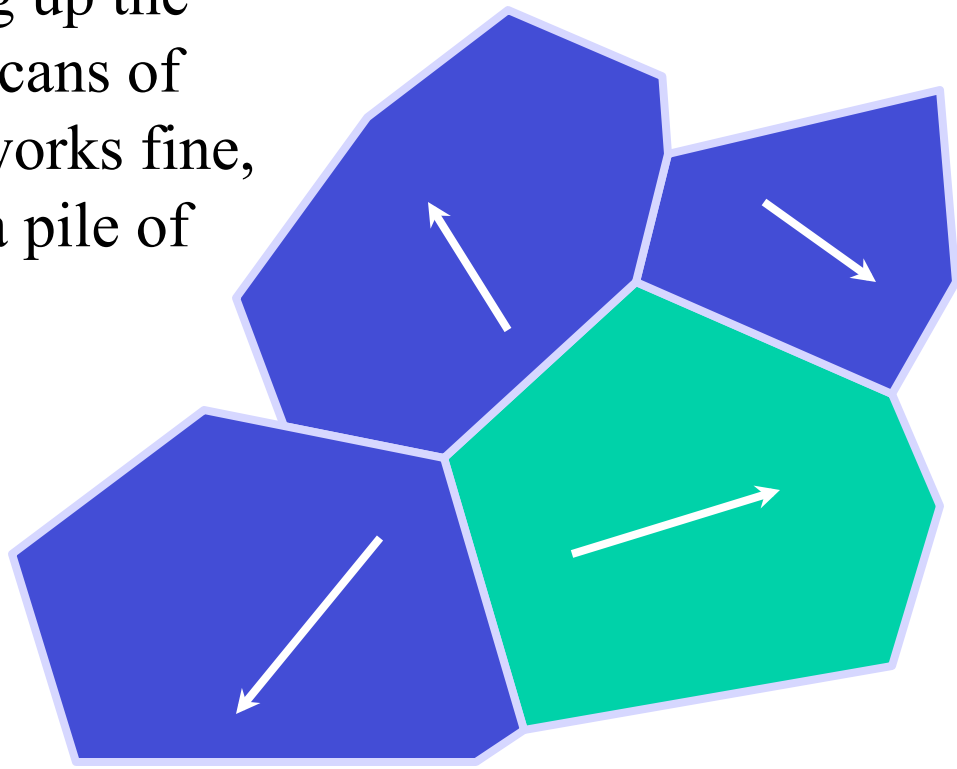
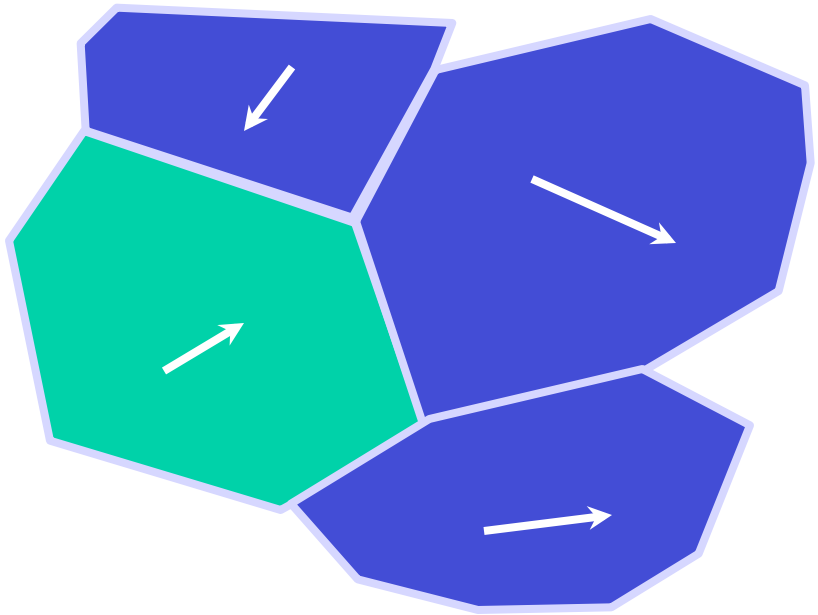
Overview: What is 3DXDM?

Polycrystalline materials are made up of single-crystal grains each with a different orientation.

Current techniques to map this microstructure involve slicing up the sample and performing 2-D scans of each exposed surface. This works fine, but the sample is reduced to a pile of dust (so it's not repeatable)

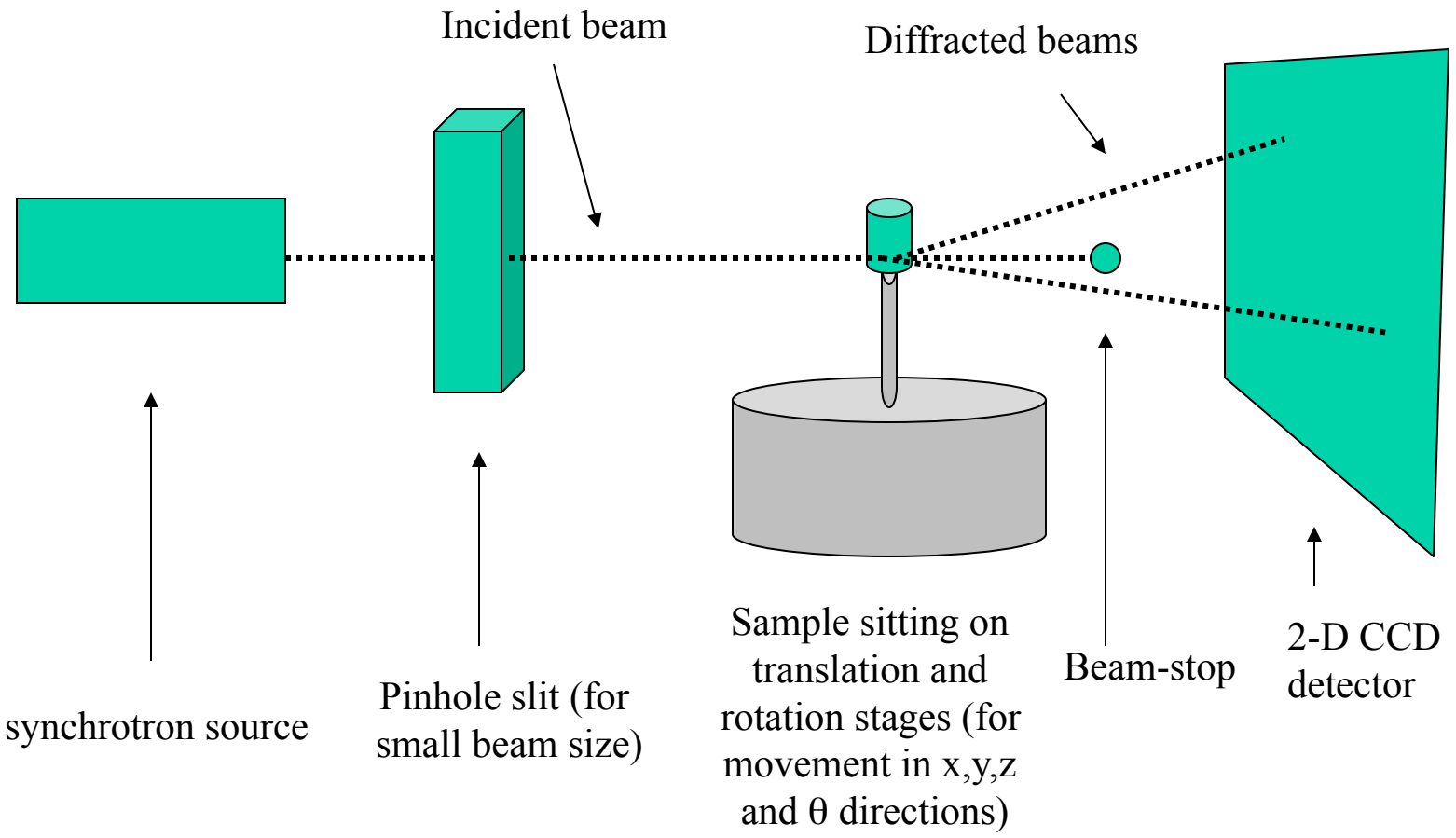


However, using an intense X-ray beam combined with some clever data-taking and analysis, it appears to be possible to do the same thing without destroying the sample. This is the gist of 3DXDM.



If this technique works well, it would be advantageous because it would allow the study of grain-boundary dynamics. For example, one could map a set of grains in a sample, then anneal it or stress it in some way. Afterward, the sample could be rescanned to see changes.

Experimental Setup:



Method:

STEP 1: Find the orientation of the grain at the center of the rotation stage:

- Place sample on rotation stage, centered in beam.
- Rotate sample by fraction of a degree intervals, recording diffraction image at each step (~0.1s counting time). Stop at 180^{O}.
- Using software algorithm, locate valid diffraction peaks. Only the central grain will have been in the beam during the full rotation, and thus will be the only one which will have a full set of peaks.
- Eliminating peaks from other grains, calculate the grain orientation of the central grain.

Method:

STEP 2: Using information from step 1, map the boundary of the central grain.

- From orientation data, pick a strong diffracted peak which is coming from the central grain
- Translate sample until the diffracted peak is at half-intensity
- Now, continue in one of two ways:
  - Follow the half-intensity contour (probe volume must be smaller than the size of one grain - requires complex optics), repeat for each “slice” of sample
  - Raster in discrete grid over the sample, rotate to another diffracted peak at a different angle, repeat and use tomographic techniques to rebuild 3D volume

Simulation/Control:

Since a large number of complex algorithms need need to be developed to handle the on-the-fly data analysis required for a 3DXDM setup, it is much less expensive to test them on a simulated apparatus.

Currently implemented features:

- ☑ X-ray optics for crystals with cubic lattices (sc, bcc, fcc)
- ☑ Basic experimental components (emitter, sample, CCD detector, rotation/ translation stages)
- ☑ Arbitrary polycrystalline samples/shapes/orientations (voxel-based model)
- ☑ Generation of random polycrystals for analysis
- ☑ Scans to produce data sets for orientation determination
- ☑ Boundary mapping scans

How it works:

Using a ray-tracing method, the path of X-rays from the source through the sample to the detector is calculated.

The sample is represented as a 3D array of cubic voxels, each of which is indexed to a grain. Each grain contains orientation and structure information from which the diffracted rays are found.

Mathematics:

$$\vec{G} = h\vec{B}_x + k\vec{B}_y + l\vec{B}_z$$

$$\vec{B}_i = R\left(\frac{2\pi}{a}\right)\hat{i}$$

G is the scattering vector, where *h,k,l* are miller indices, *a* is the cubic lattice constant, R is rotation matrix for orientation

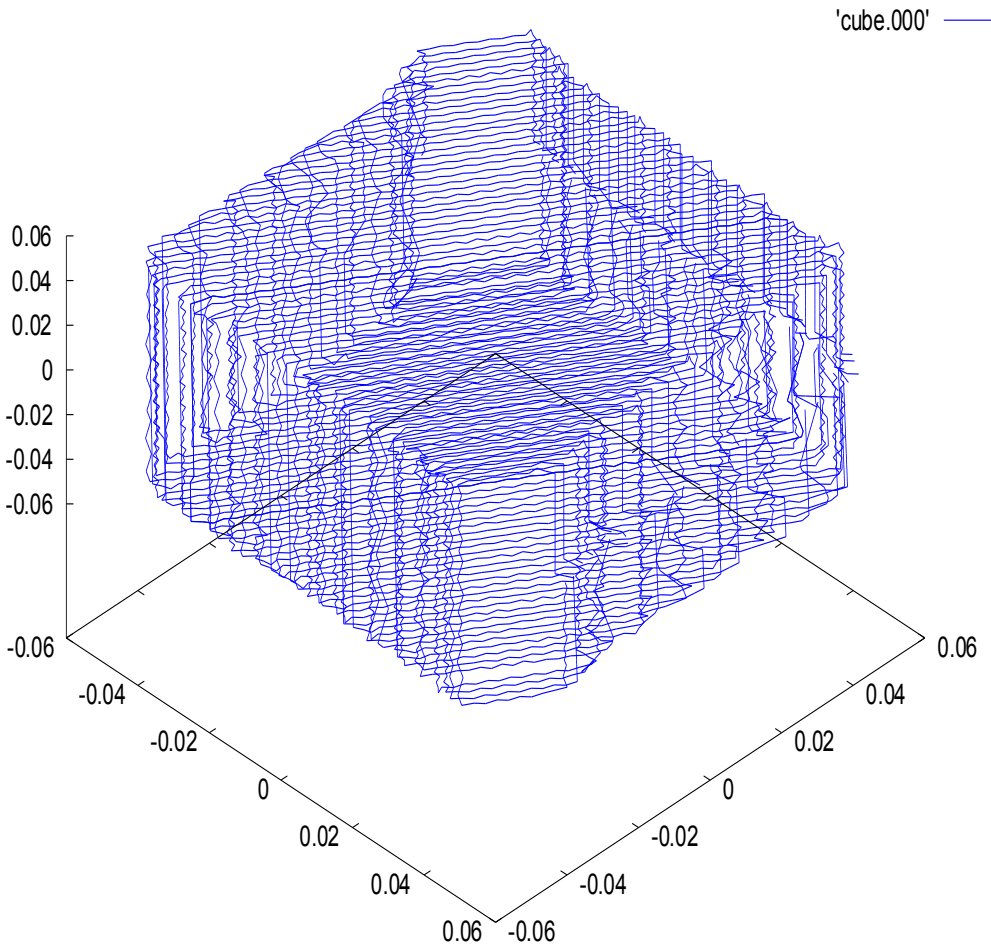
$$G^2 = -2\vec{k}_i \cdot \vec{G}$$ ←

If the condition for scattering is met,

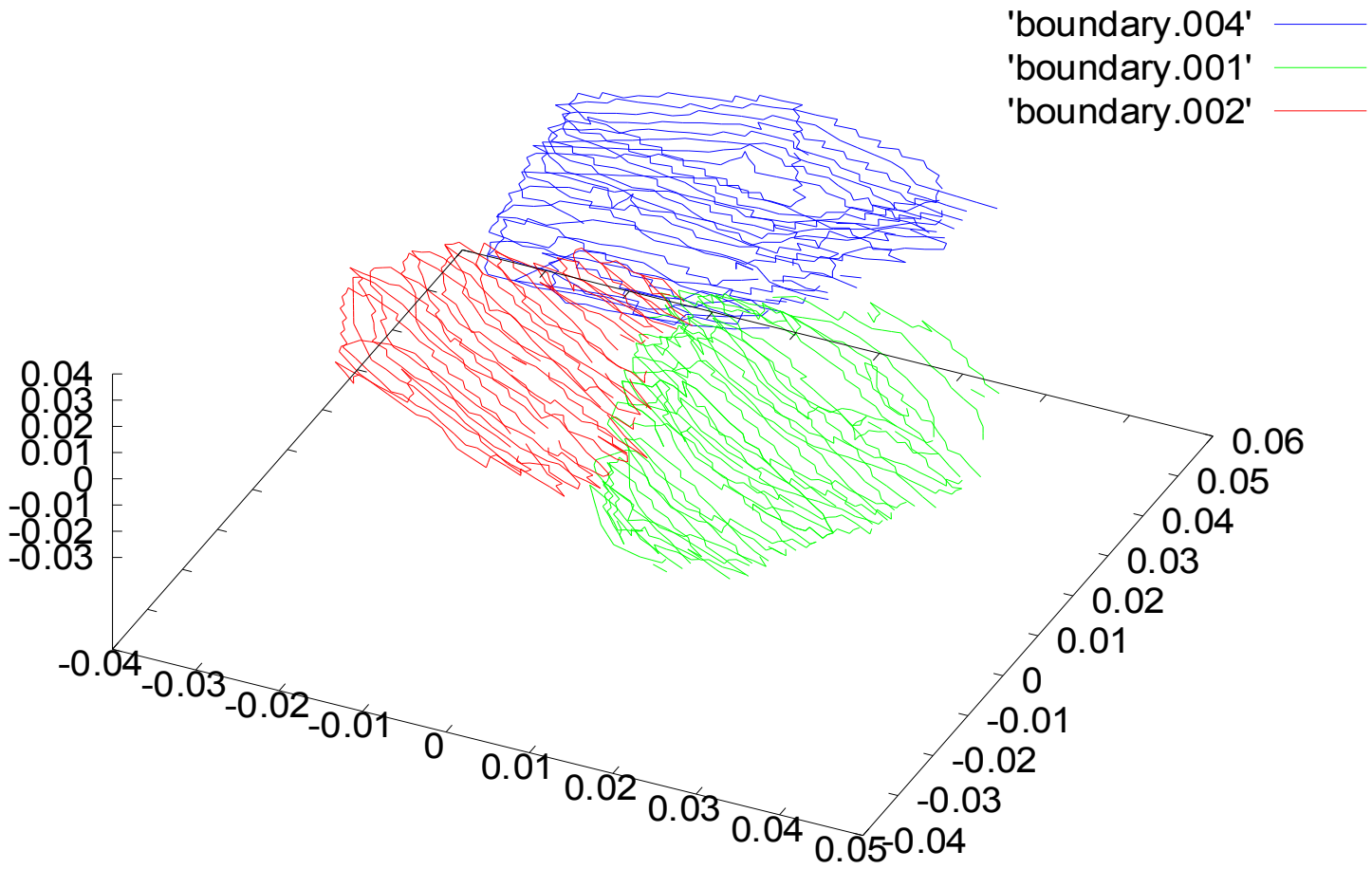
$$\vec{k}_f = \vec{k}_i + \vec{G}$$ ←

Then this is the diffracted beam direction

Output:



Test of path-following mapping algorithm on simulated cube-shaped grain.

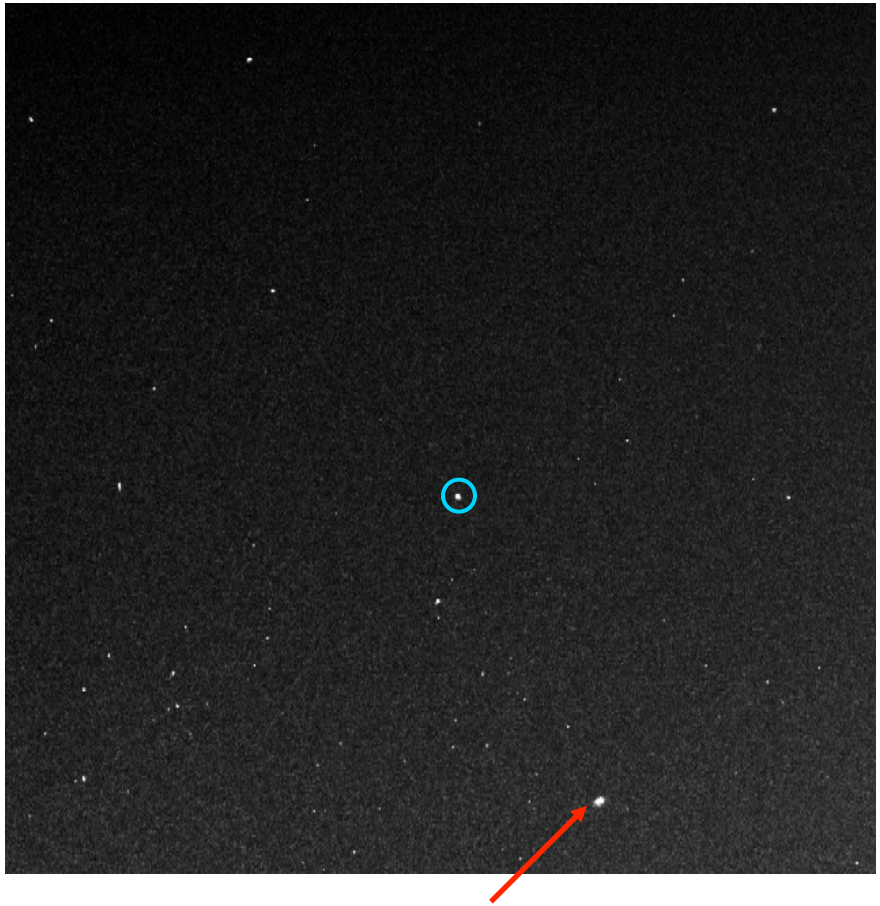


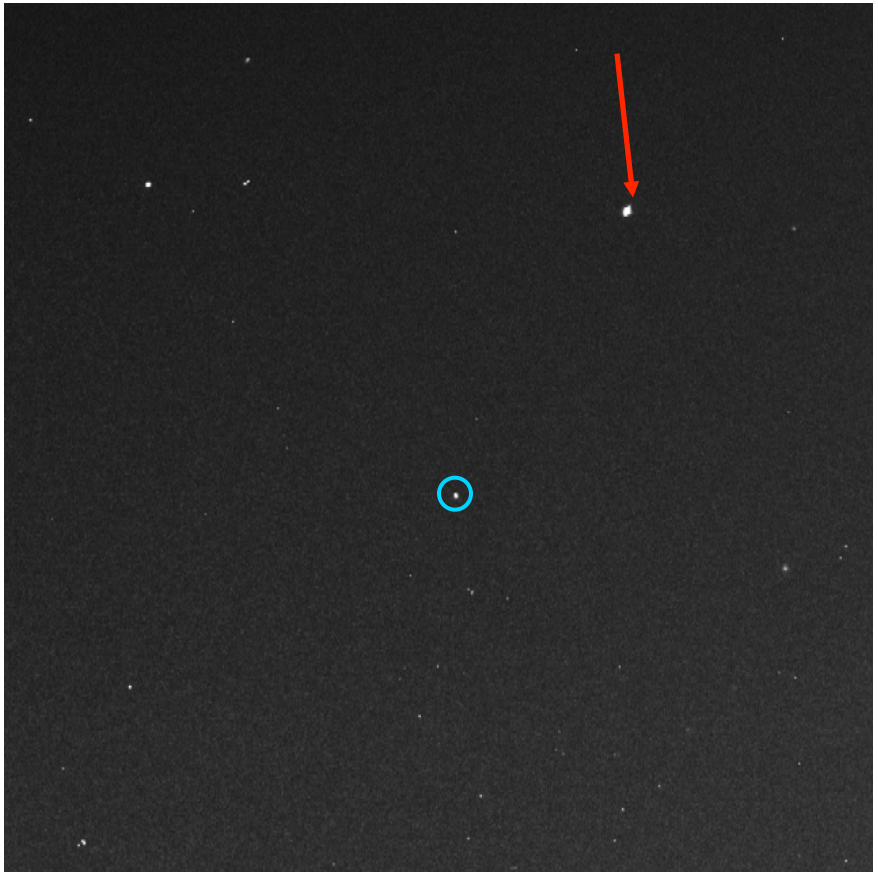
Test of path-following mapping algorithm on several simulated “realistic” grains

Overall Status:

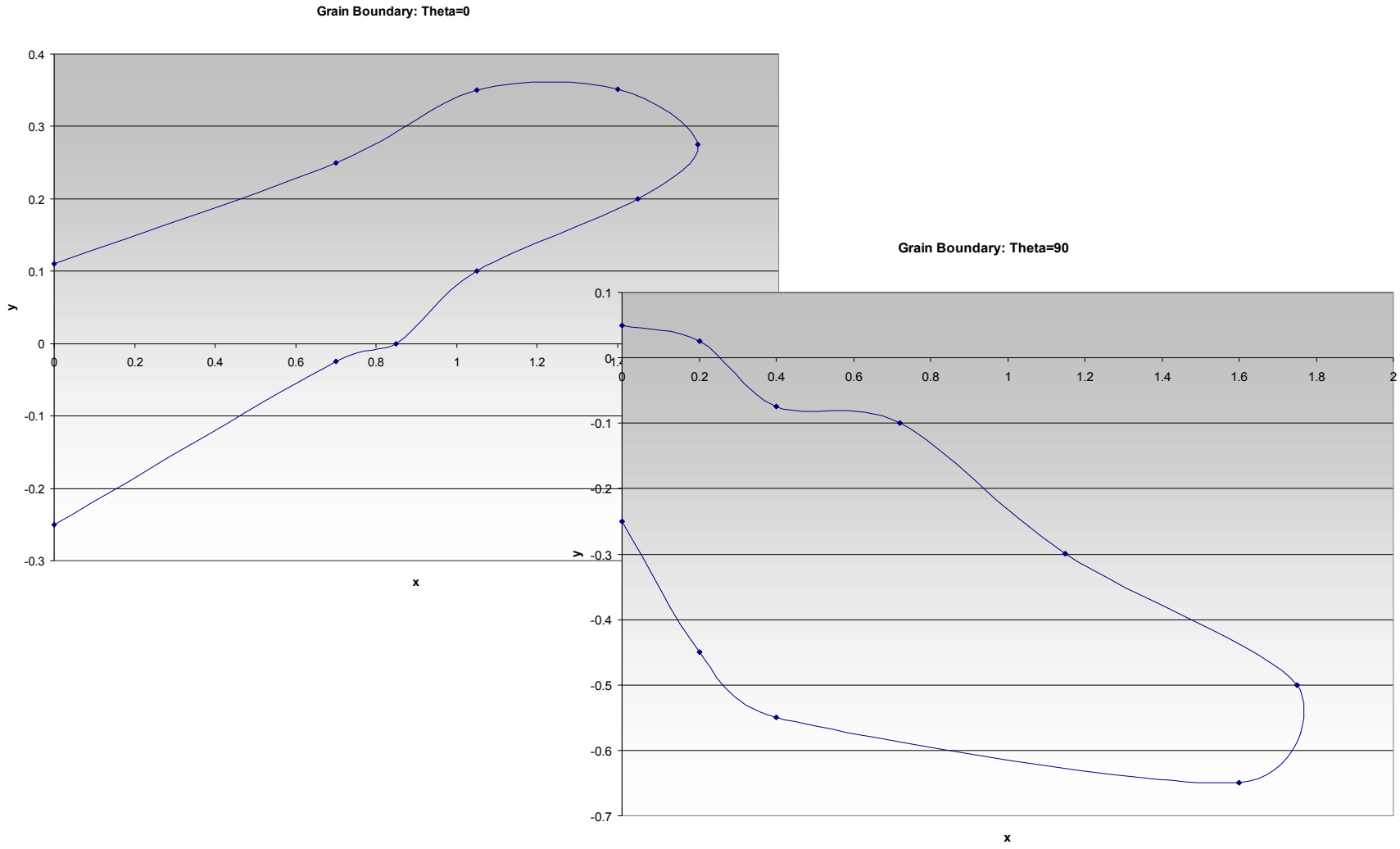
- The experiment has been computer-simulated with promising results.
- Software for orientation determination has been written and tested with simulated data.
- Basic algorithm for efficient grain boundary mapping has been developed.
- Initial experiments have been performed at CHESS synchrotron demonstrating the feasibility of the methods.
- Prototype setup with dedicated beamline exists at Risø National Lab in Denmark

Preliminary Results:





Images of diffraction peaks at different angles for a large-grained aluminum sample (taken CHESS synchrotron facility, Cornell University). Red arrows indicate diffraction peaks, and blue circles show the incident beam.



Plots of grain boundary data (taken by hand) of aluminum sample. In the second plot, the sample was rotated 90^{O} about the z-axis.

Future Challenges:

(for somebody other than me)

- Hardware setup, accuracy of translation/ rotations
- Interfacing hardware (detector, translation stages, etc.) with software to automate system
- Data reduction software (partially complete)
- Method for limiting beam length for 3D mapping w/o tomography