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Quantitative Texture Analysis Via **TILT-A-WHIRL** and **MAUD**

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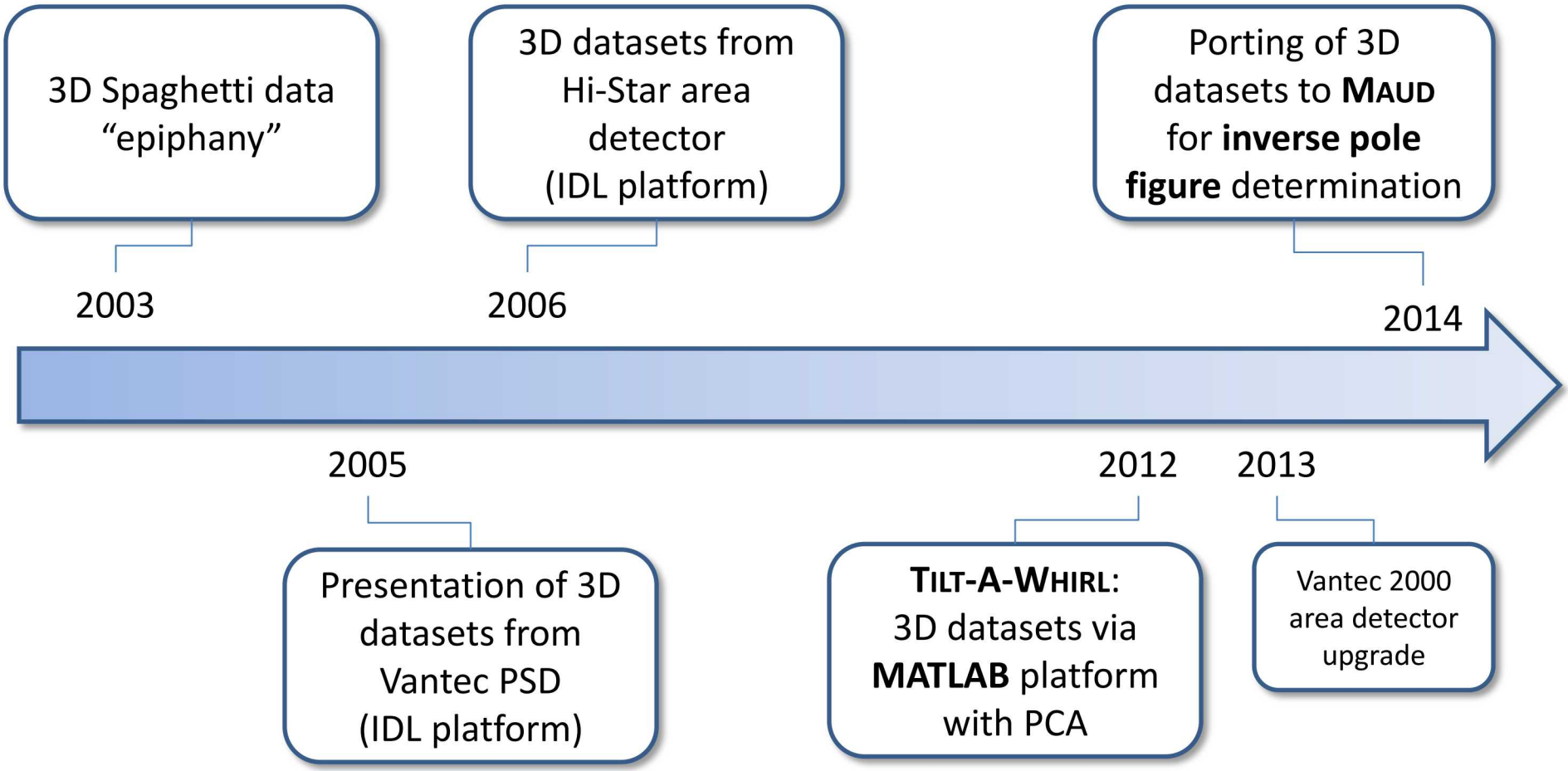
Development Timeline



Three-dimensional Interactive Data-Language pole figure visualization

By: Frazer, CS; Rodriguez, MA; Tissot, RG
Conference: Denver X-Ray Conference Location: Colorado Springs, CO Date: AUG 01-05, 2005
POWDER DIFFRACTION Volume: 21 Issue: 2 Pages: 102-104 Published: JUN 2006

TILT-A-WHIRL: a texture analysis package for 3D rendering of pole figures using MATLAB

By: Rodriguez, Mark A.; Pearl, Megan R.; Van Benthem, Mark H.; et al.
POWDER DIFFRACTION Volume: 28 Issue: 2 Pages: 81-89 Published: JUN 2013

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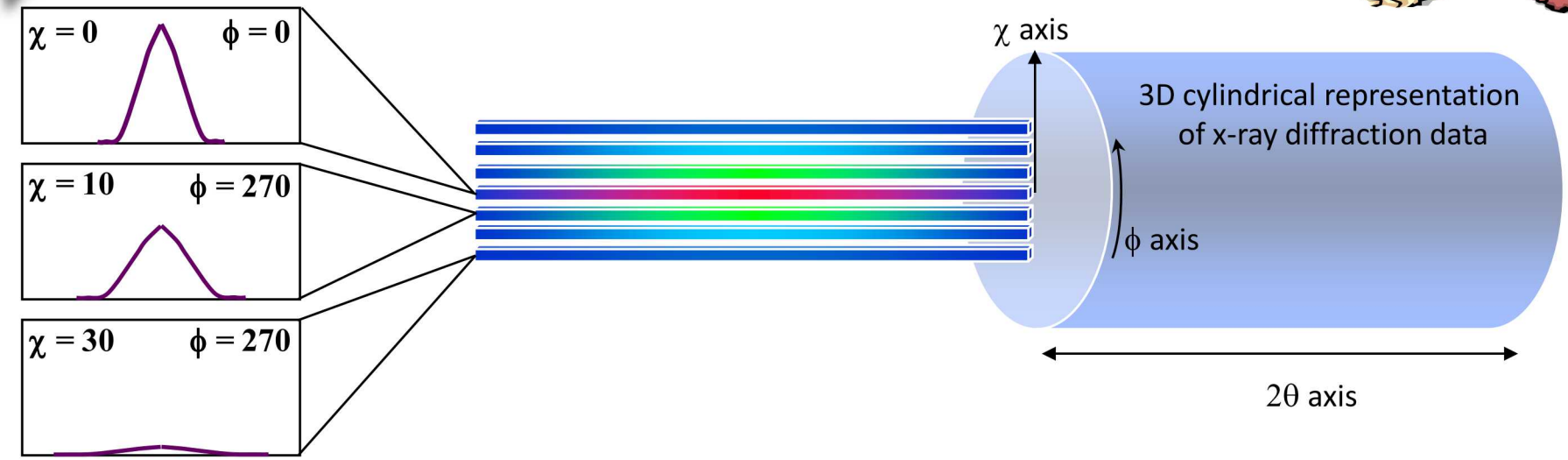
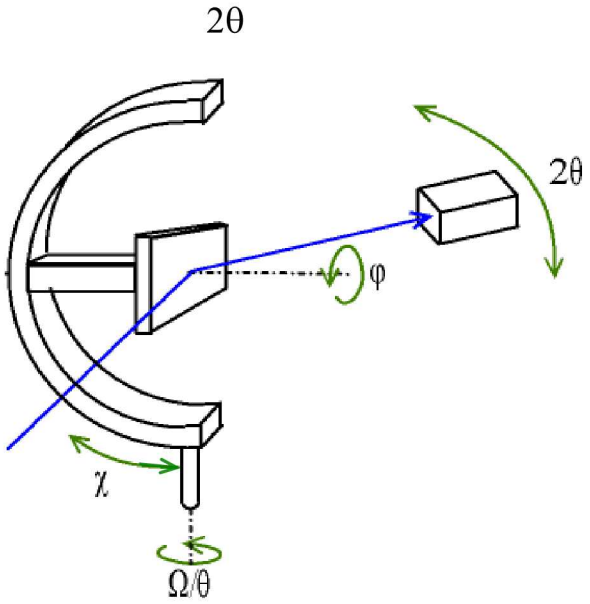
What is Spaghetti data?





- Generate θ–2θ scans with assigned/fixed (χ) and (ϕ) pairings.
- Three dimensional dataset (2θ, χ, ϕ) with scalar (color) intensity.
- θ–2θ scans are converted to line with color intensity variation.
- Pole figures can be created by slicing 3D dataset.

Christophe Dang Ngoc Chan - wikipedia

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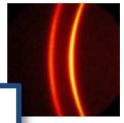
Data Processing and Analysis Flowchart for **TILT-A-WHIRL**

batch.slm

Data collection via slam file

*.gfrm



3360 frames
20 hrs / 13 GB

integration.slm

Frame integration and file stitching

*.PLT

840 merged ASCII files
13 MB

Matlab.exe

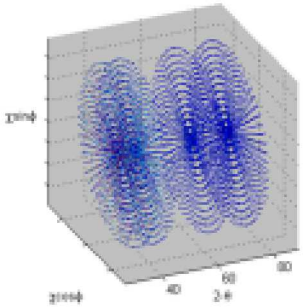
File clean-up by **Spaghetti/ Linguine**

*.rft

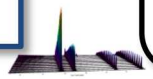
840 2θ scans with χ,ϕ tags "spaghetti data"

Matlab.exe

Pole Figure Explorer for visualization and plotting



*.rft

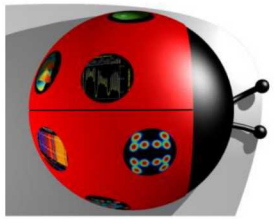


14 raw data ϕ-merged 2θ scans

Jade.exe

Phase ID, LP refinement, macro-strain via **JADE**

*.esg



MAUD
Material Analysis Using Diffraction

Full Rietveld structure refinement with texture

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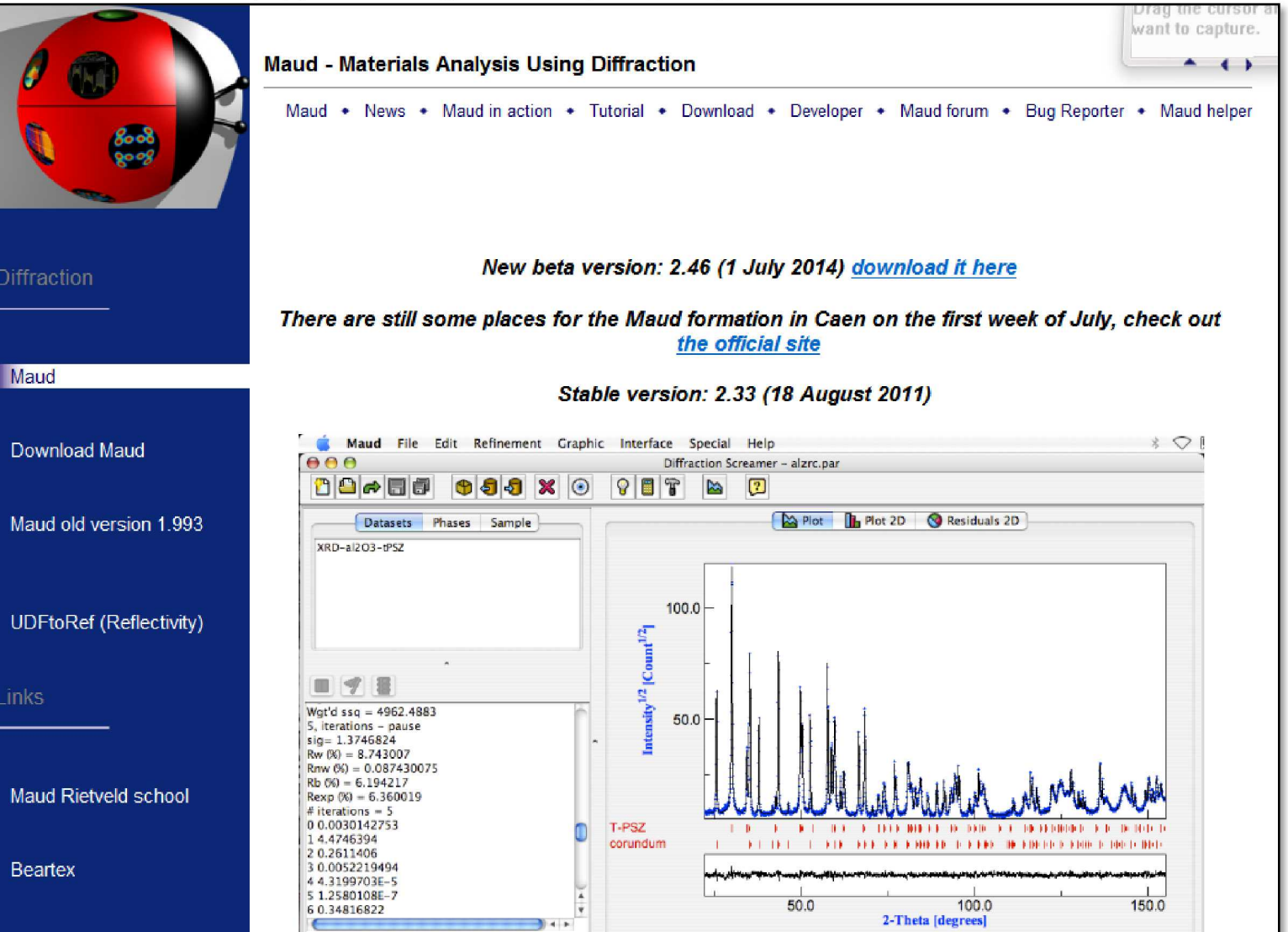
The MAUD software package is “one-stop shopping” for refinement of structural parameters, microstructural details, and texture of large diffraction datasets.

http://www.ing.unitn.it/~maud/



Author:
Dr. Luca Lutterotti
Univ. Trento

Collaborator:
Dr. Rudy Wenk
UC Berkeley

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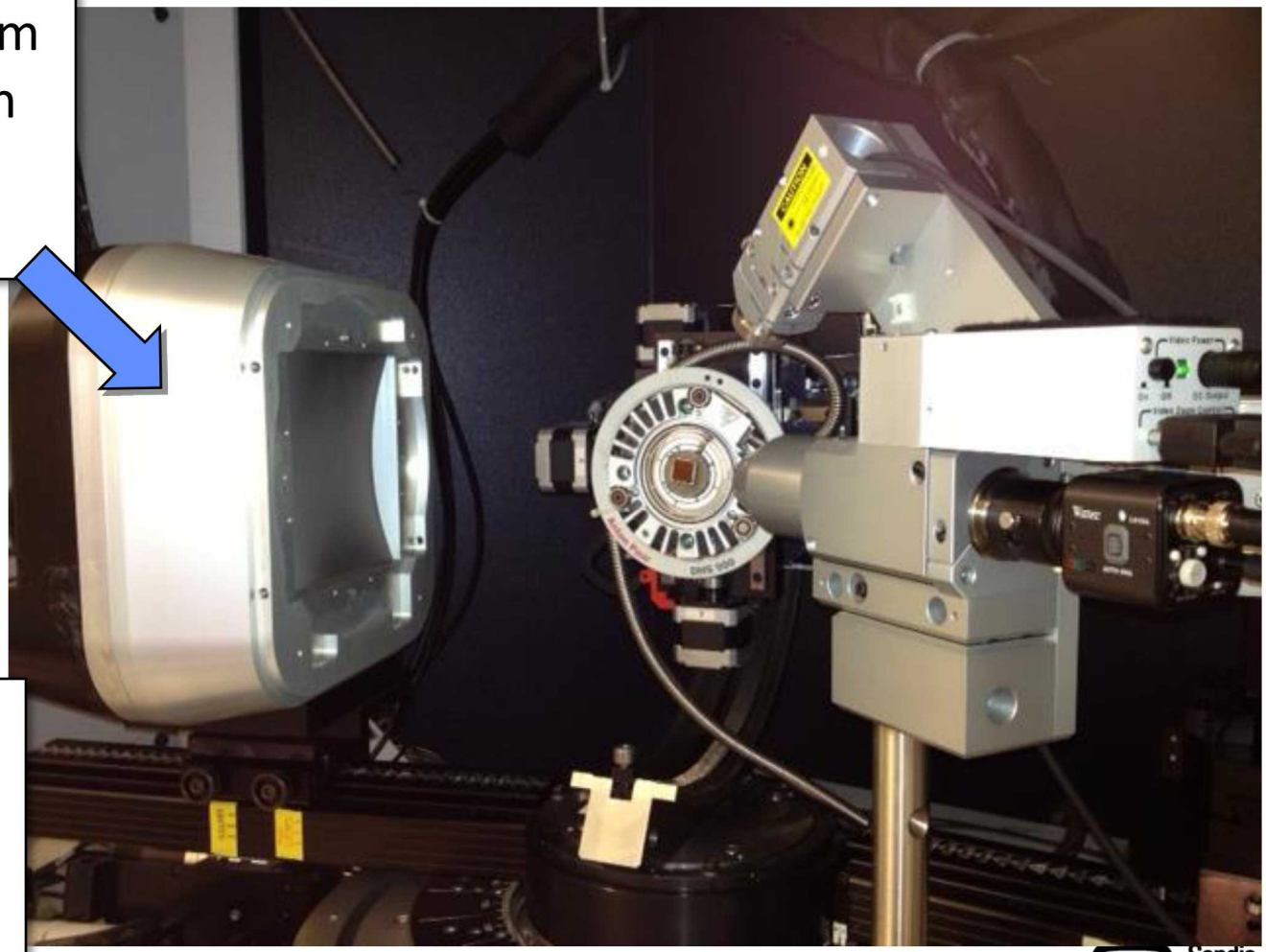
A recent upgrade of our Bruker D8 diffractometer to a Vantec-2000 area detector has reduced issues related to detector damage.

Bruker D8 System configured with Vantec-2000 Area Detector



Now we can run strongly diffracting samples or film samples on single crystal substrates.

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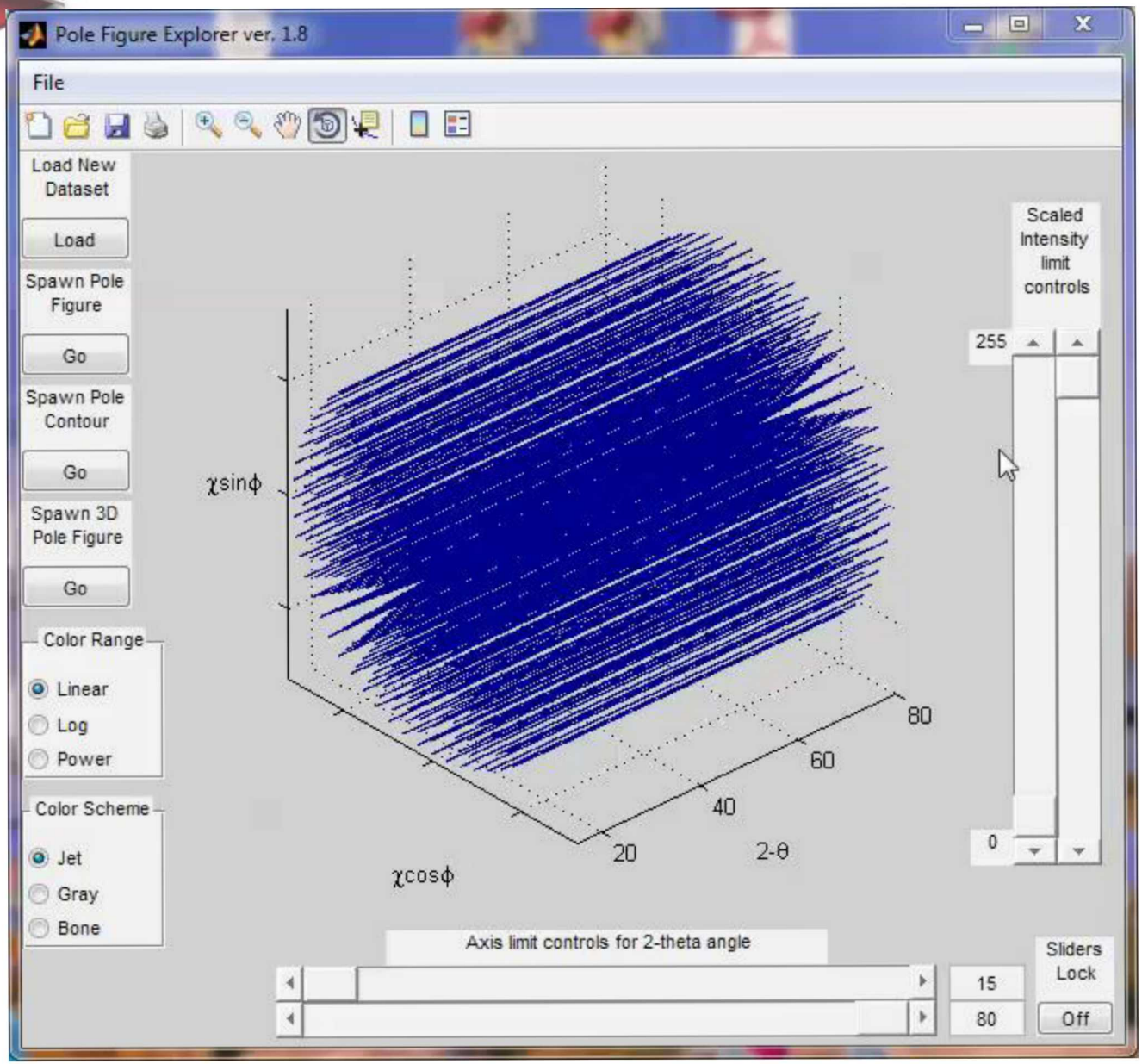
POLE FIGURE EXPLORER video



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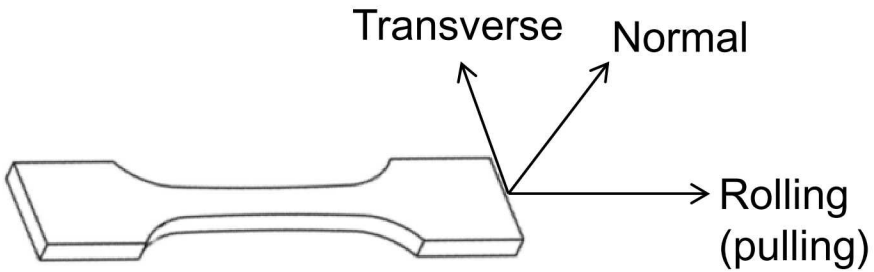
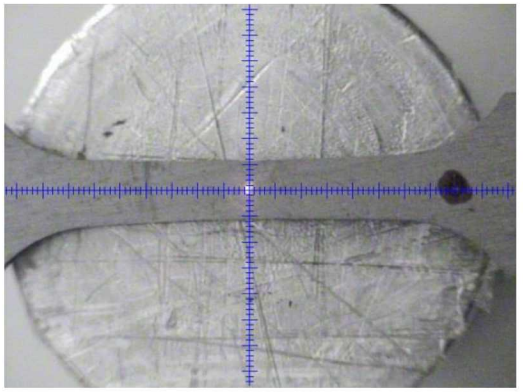
We have applied **TILT-A-WHIRL** w/ **MAUD** to analyze single crystal Ta dogbone samples

- **1st Problem**: Can we determine/verify crystallographic orientation of dogbone samples prepared for mechanical testing?



Schematic of dogbone orientation during XRD data collection



Picture of dogbone sample on diffractometer

- **2nd Problem**: Can we detect grain ***re-orientation*** after mechanical testing (pulled condition)?
- We will analyze the same bar in the as-received (i.e. unpulled) state and after mechanical testing (i.e. pulled).

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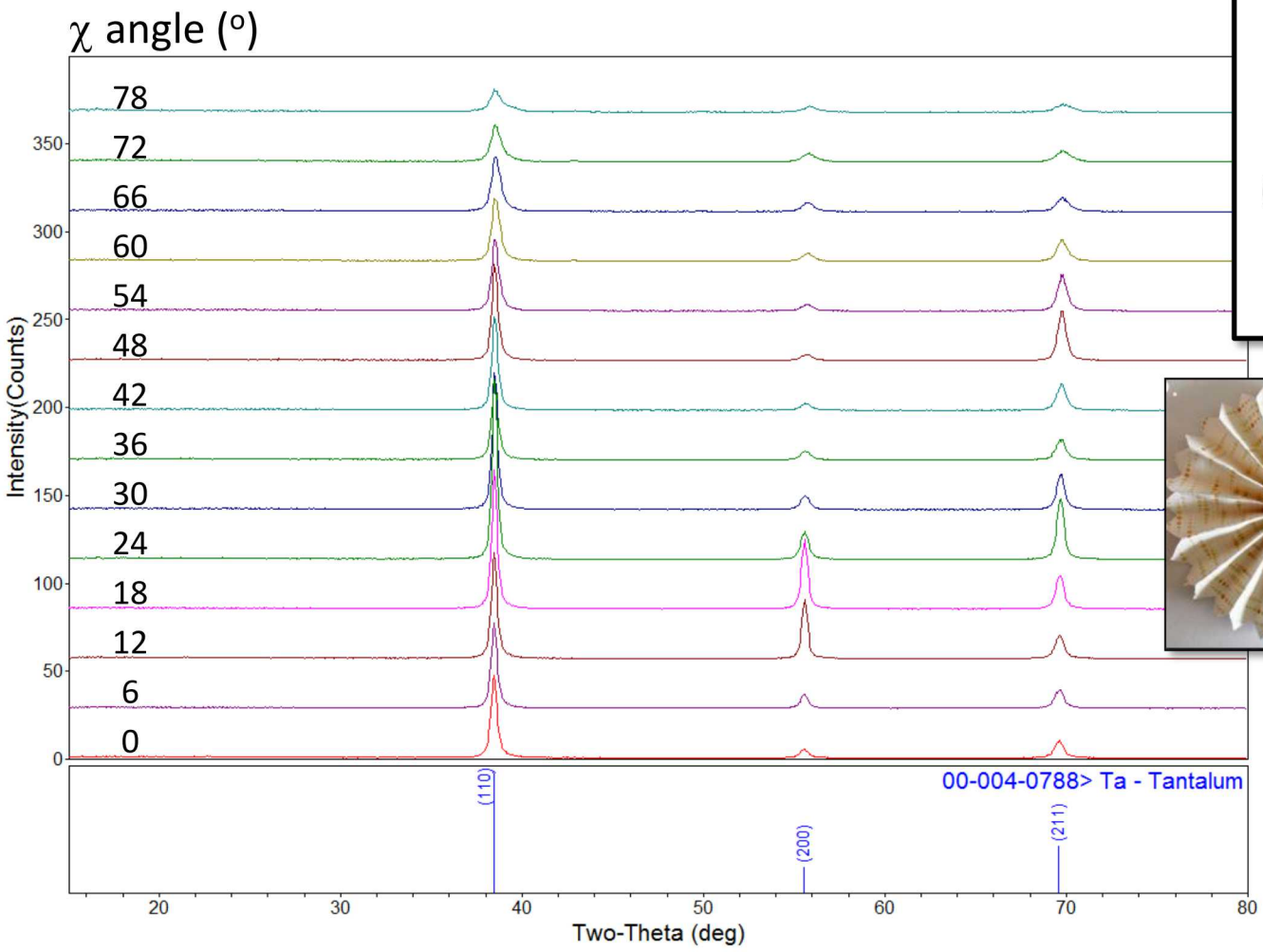
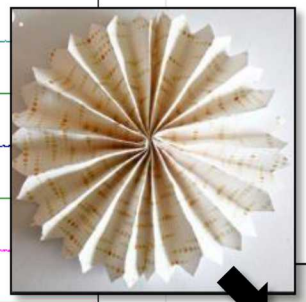
A quick look at the ϕ-merged data for the unpulled dogbone indicates strong texture.



Each χ scan represents the integrated intensity (merging) of the 60 ϕ scans at a given χ angle



This is similar to the collapsing of an accordion pinwheel flower

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Initial pole figure results for “unpulled” dogbone plotted via **POLE FIGURE EXPLORER**

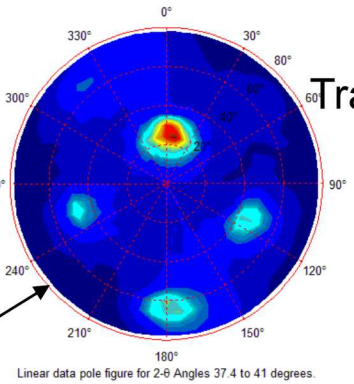
Raw pole figure: 2D color contour

Rolling (pulling)

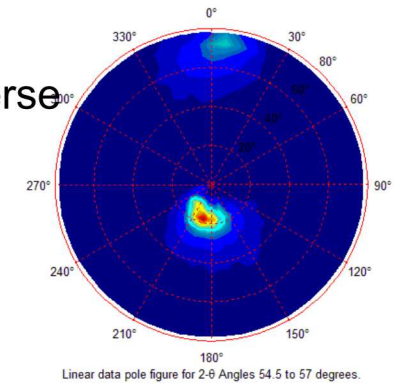
Transverse

Pole figure boundary limited to $\chi = 78^{o}$ (i.e. <90°)

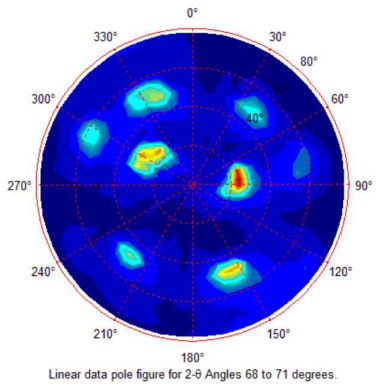
110



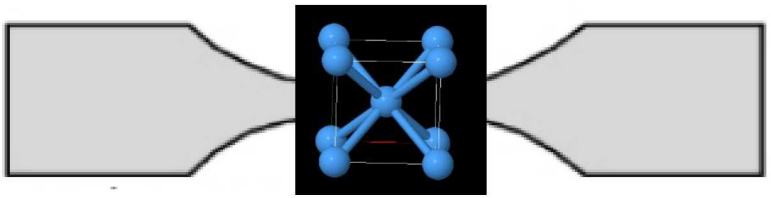
200



211



Rolling (pulling)



Transverse

- Observation of isolated intensity with bi-axial nature is consistent with single crystal diffraction.
- Broadened spots are likely a consequence of large (6°) step-size in χ, ϕ along with omega scanning during data collection.
- Initial qualitative assessment of texture suggests that the BCC Ta a-axis is tilted from the normal direction of the bar.

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MAUD refinement

- Spaghetti data were converted to *.esg format files and read into **MAUD** software.
- Data above χ = 60^o tilt angle often show significant peak broadening due to defocusing.
- We employed only a portion of spaghetti dataset from χ = 0, 6, 12…54^o tilt angle (ϕ = 0, 6, 12…354^o) for a total of 600 θ-2θ scans. This resulting dataset contained 780,000 observations.
- We report data for a [100] or “a-axis” oriented dogbone **before** and **after** pulling.

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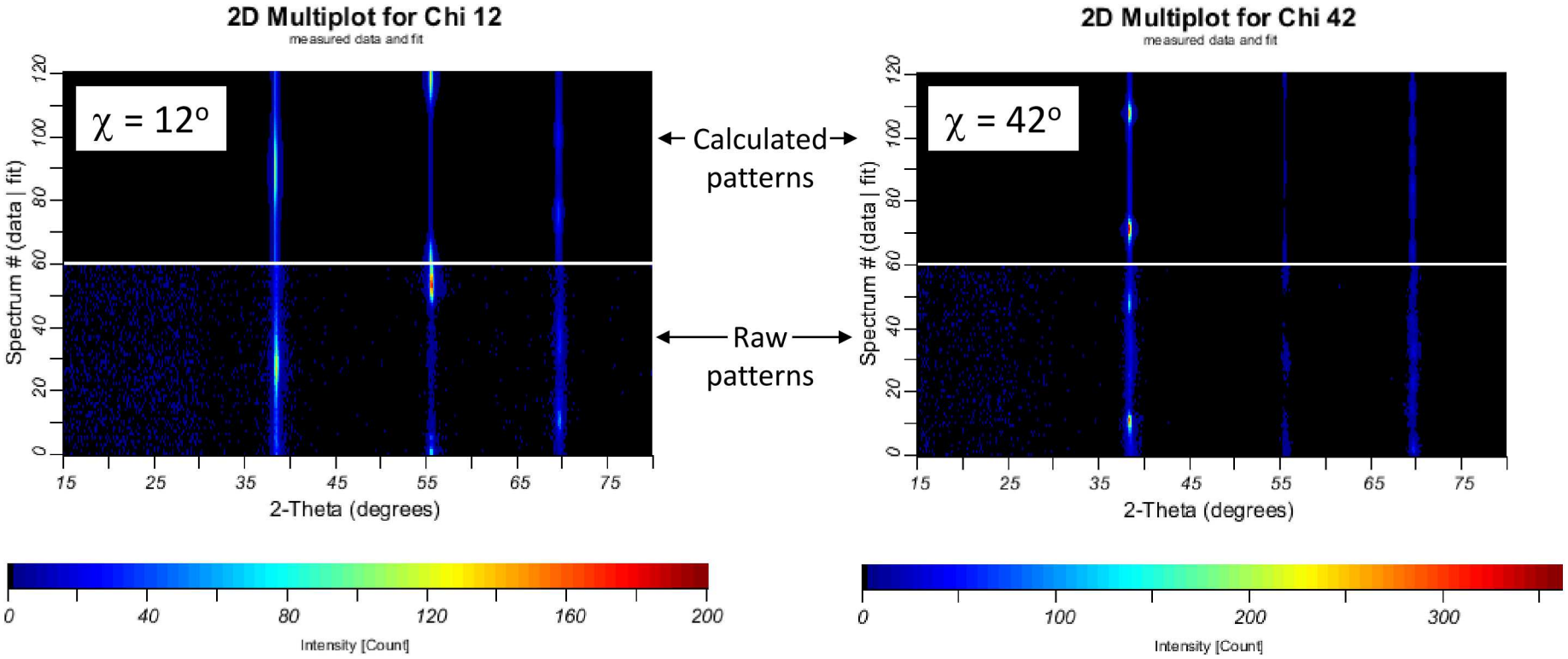
MAUD generates 2D contour plots of raw vs calculated patterns to evaluate refinement



- These data for the **pulled** dogbone show appropriate predication of peak intensity between raw and calculated patterns, indicating that the refinement is properly tracking the texture behavior.
- Two datasets are shown: $\chi = 12^{o}$ and $\chi = 42^{o}$. All 60 ϕ scans are plotted for each χ angle.

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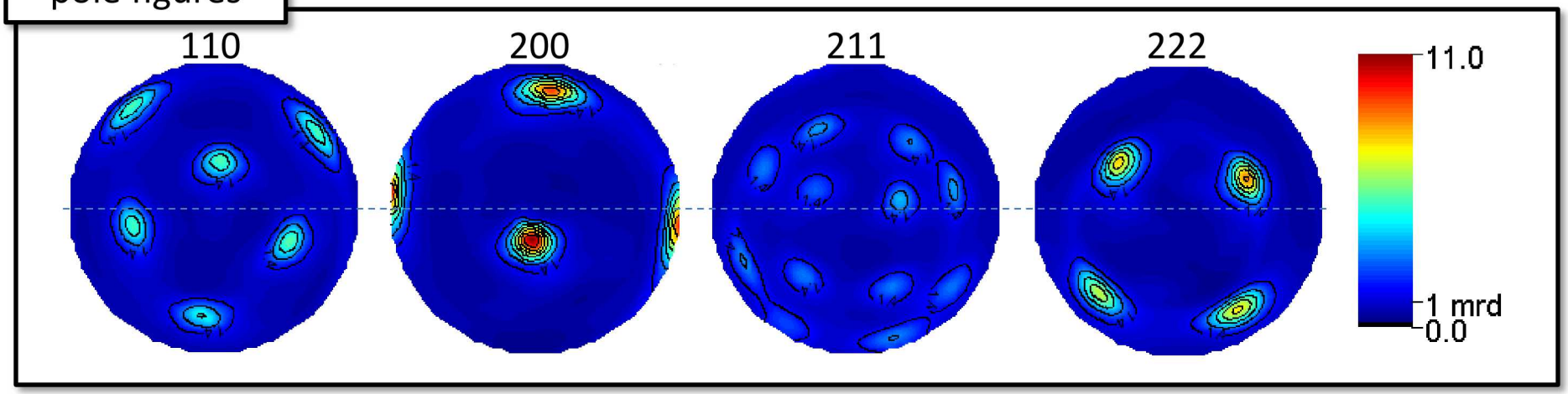
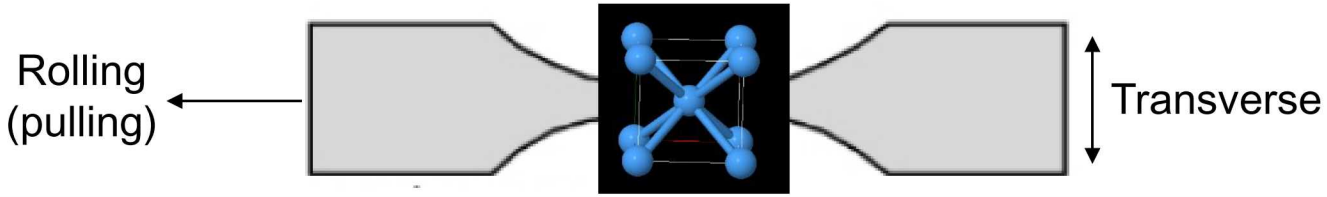
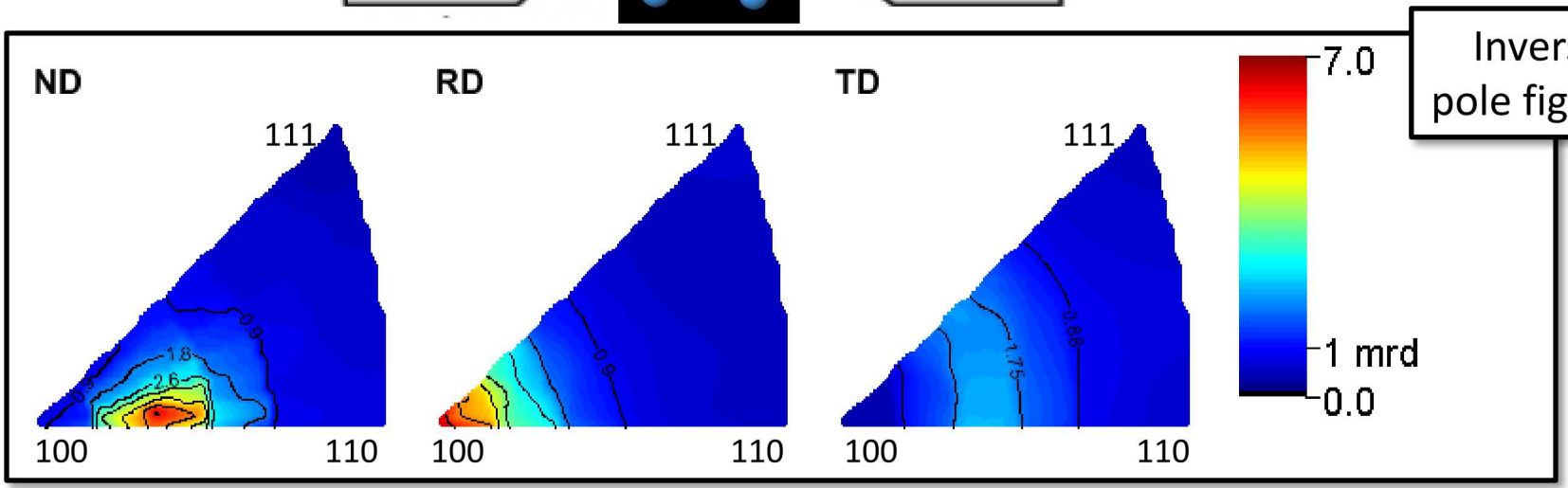
Reconstructed pole figures and inverse pole figures for the **as-received (unpulled)** dogbone show near coincidence of the Ta a-axis with rolling direction.

Reconstructed pole figures





Inverse pole figures



2013-3791

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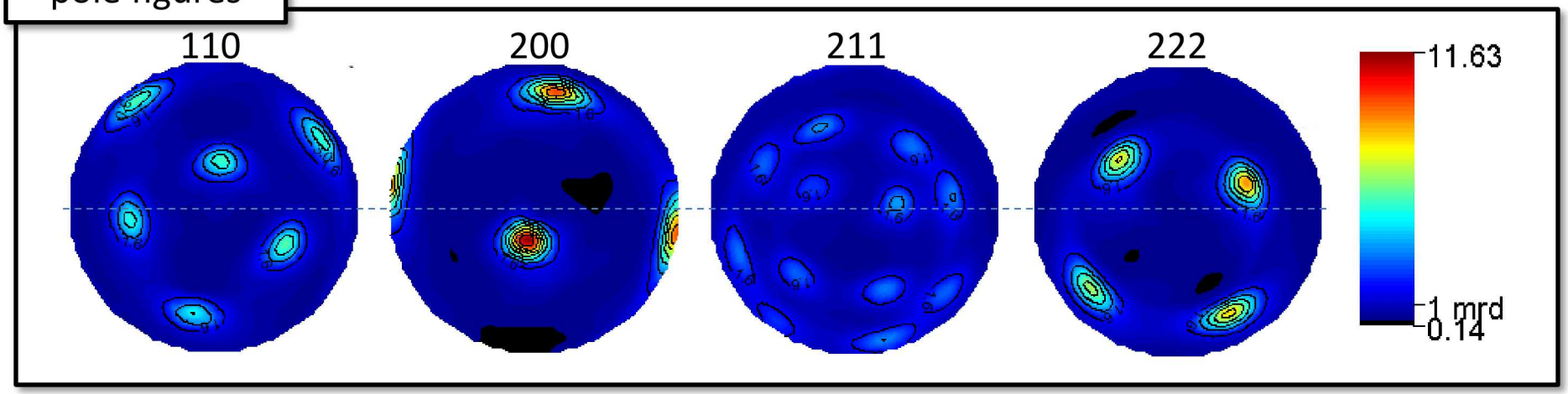
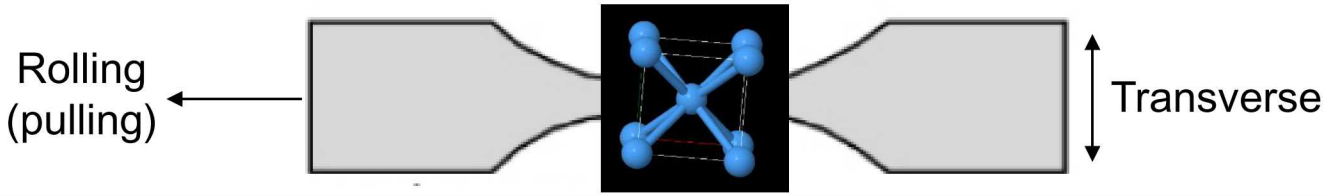
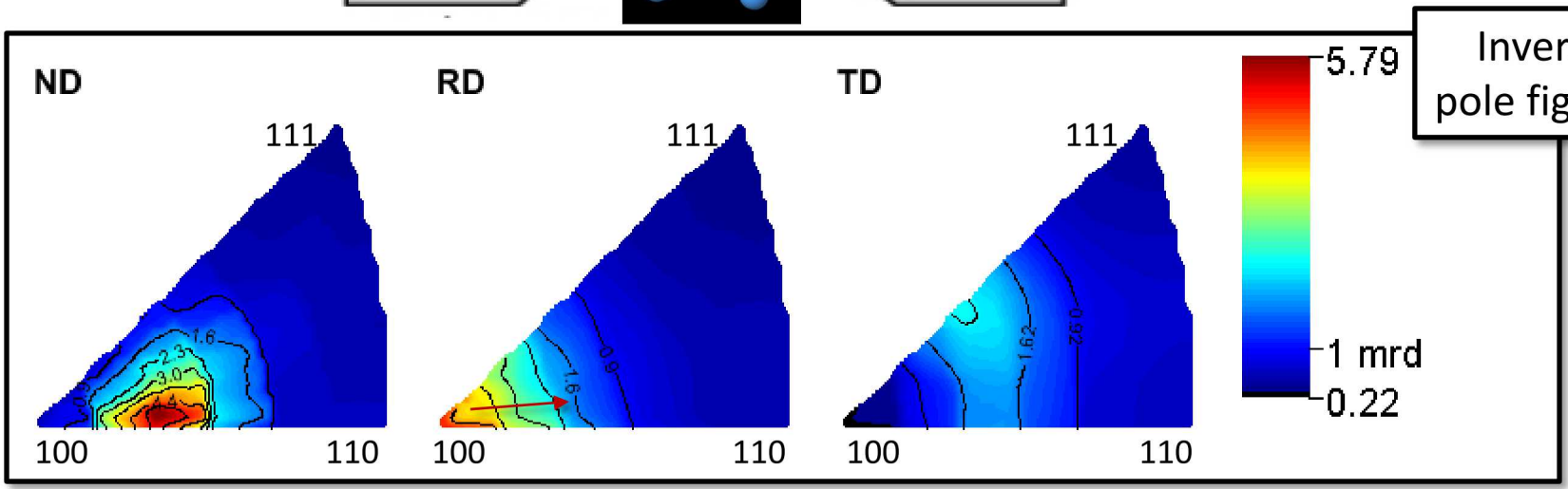
Reconstructed pole figures and inverse pole figures for **pulled** dogbone indicates Ta grain rotation to transition from [100] to [110] along pull direction.

Reconstructed pole figures





Inverse pole figures



2014-5104

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Summary

- We have successfully extended **TILT-A-WHIRL** texture analysis to include quantitative texture determination (inverse pole figures) through the use of **MAUD** software.
- We can employ **TILT-A-WHIRL** w/ **MAUD** to diagnose/verify crystallographic orientation of single crystal dogbone specimens.
- Strain causes rotation of grains from [100] preference along rolling direction toward [110] which is consistent with other analyses and modeling/simulation.
- Future work: couple XRD, EBSD, and modeling to predict effects of strain on texture and ultimate impact on failure.

Acknowledgment

- Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.
- The authors are grateful to Tom Buchheit (SNL) for his useful input for this work.

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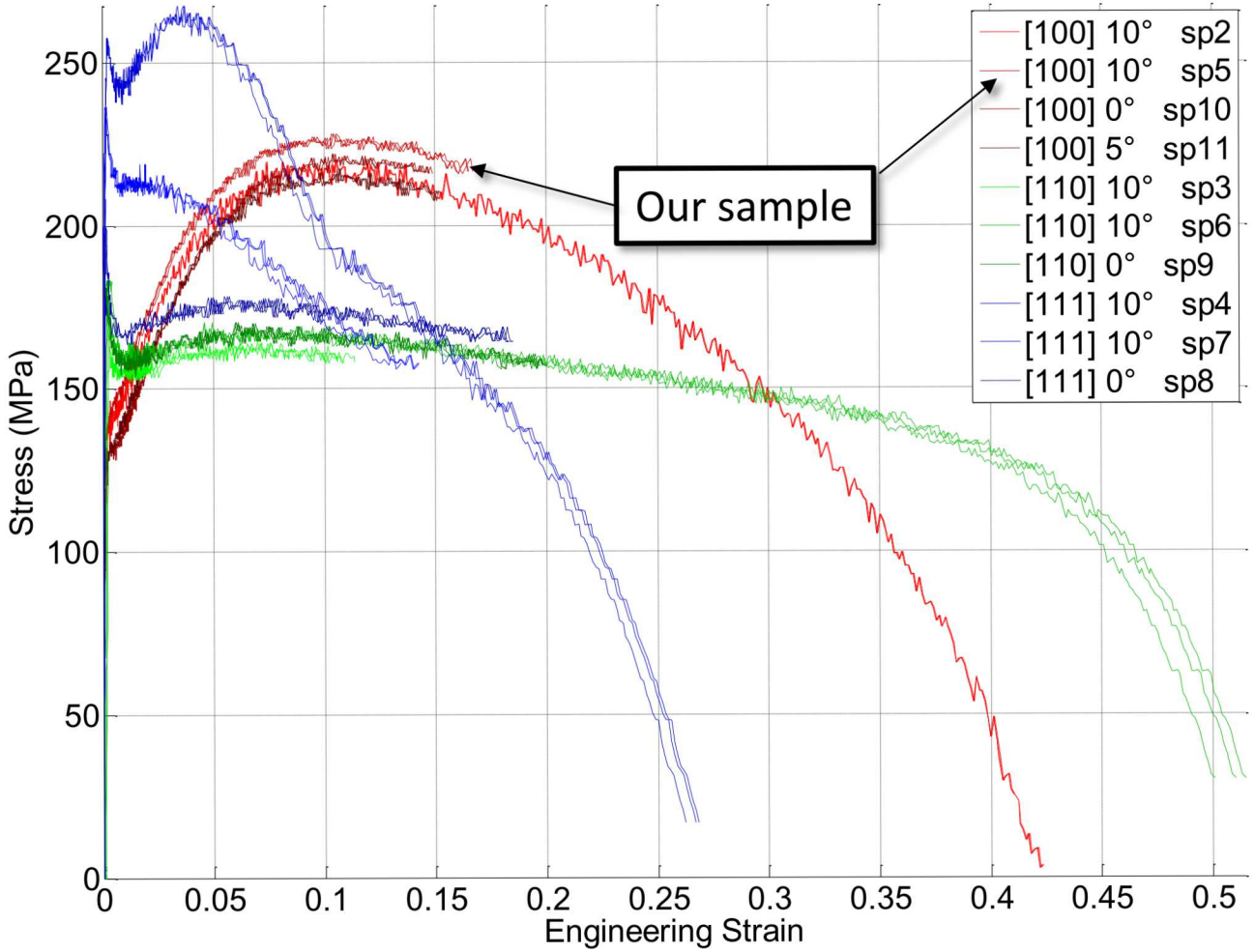
Stress strain curves for various Ta dogbone samples



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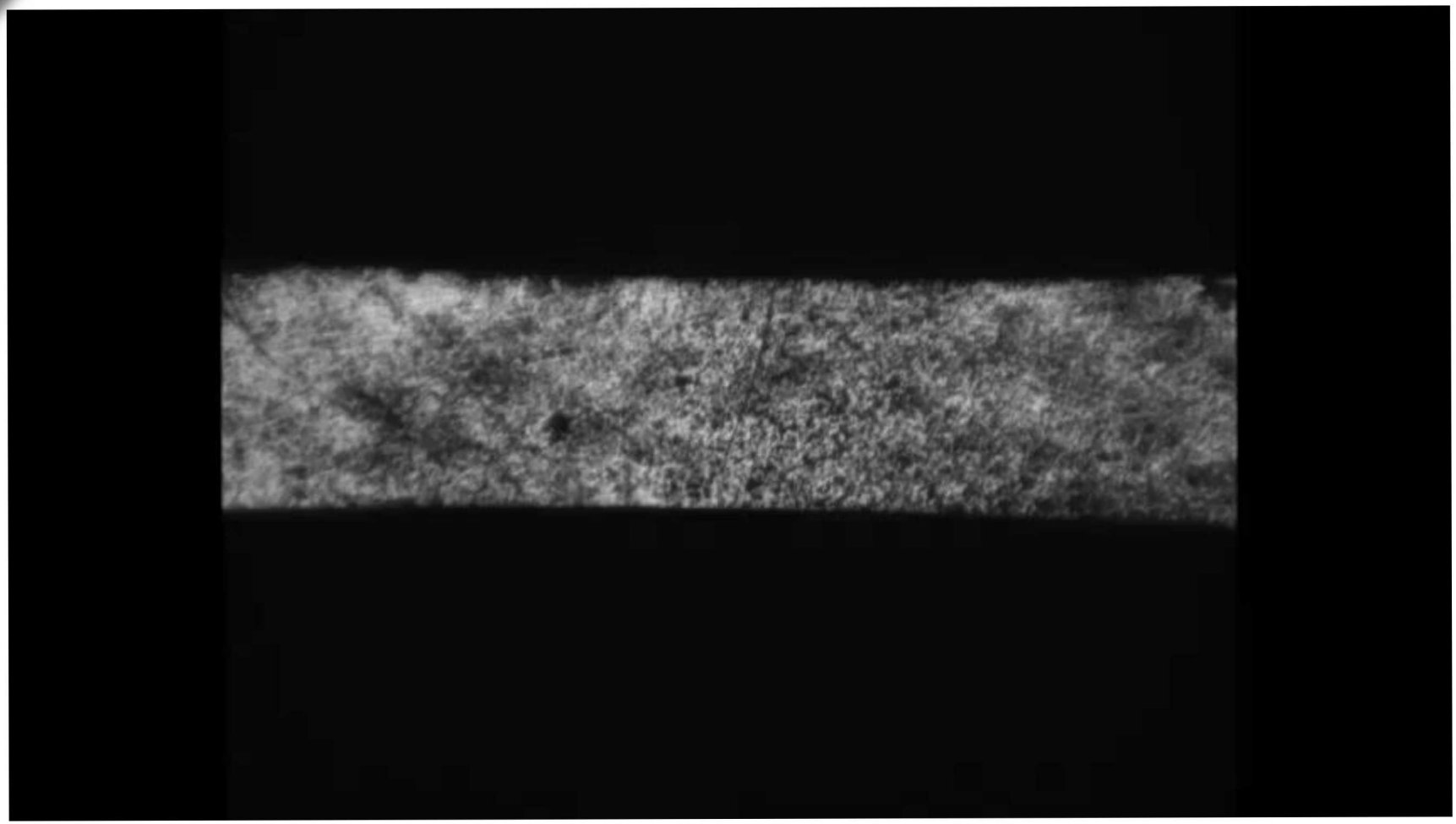
In-situ strain video



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EBSD was employed to confirm texture results obtained from analysis using **TILT-A-WHIRL** and **MAUD**

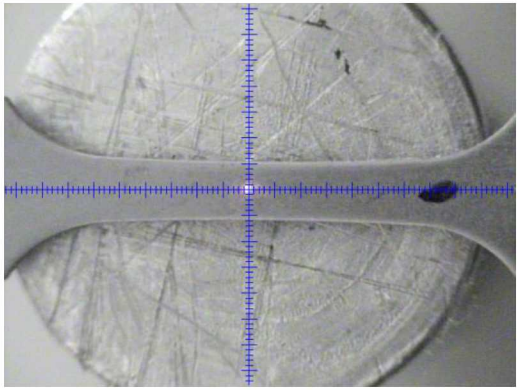
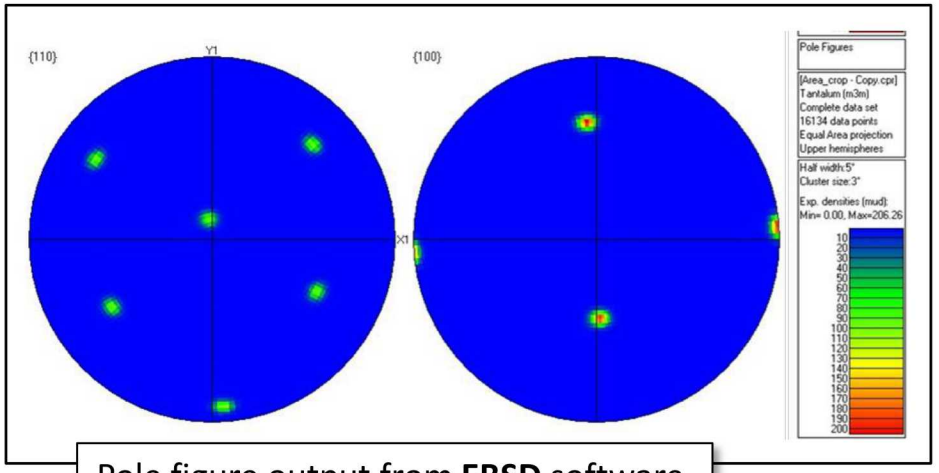
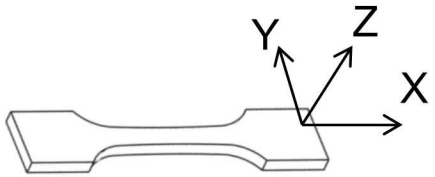


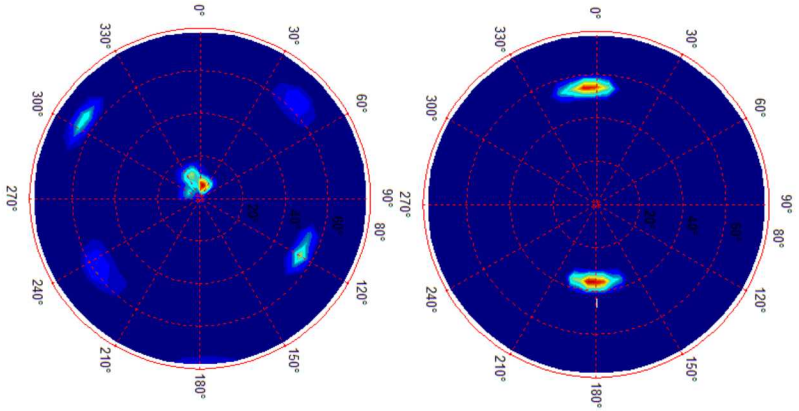
Image of sample on D8 X-ray diffractometer



Pole figure output from **EBSD** software



Schematic showing sample orientation during EBSD data collection



Pole figure output from Pole Figure Explorer

Pole figures rotated 90° clockwise match EBSD output

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