CANFSA

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

A new tool (microscope) for 3D characterization of orientation at the mesoscopic length scale

"Sight Through Sound"

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DURIP N00014-17-1-2294

Iowa State University · Optics & Photonics, University of Nottingham · University of Nottingham UK | China | Malaysia · Office of Naval Research Science & Technology · cpi coherent photon imaging

Baseline: this is a microscope

Leeuwenhoek's work defined our definition: *To see small*…

- A more complete technical description of a microscope is:
  - An instrument which
    - uses optics to direct a beam of EM radiation to
    - a specimen that is of interest where
    - the incident waves are modified by certain characteristics of the specimen
    - and for which the modified waves/particles are directed to a detector for analysis (with it's own signal modification)

Let's test this

		Optical (including Leeuwenhoeks)
Optics	Uses optics to direct a beam of EM radiation to	Light (coherent or incoherent)
Specimen Damage	a specimen that is of interest where	✔
Image Theory	the incident waves are modified by certain characteristics of the specimen	Reflection Transmission
Detectors	and for which the modified waves/particles are directed to a detector for analysis	✔ (from an observer to detectors)

SRAS is a microscopy technique that directs energy impulses (typically supplied by a laser) to the specimen, and which are sufficient to interact and generate acoustic waves (of varying modes) which can be measured using sophisticated detectors.

Interestingly, this structure (optics, specimen/damage, theory, detectors) are where most microscope developers and users spend all their time.

Spatially Resolved Acoustic Spectroscopy

Motivation for the work - *Sometimes, we need data and statistics as the mesoscale!*

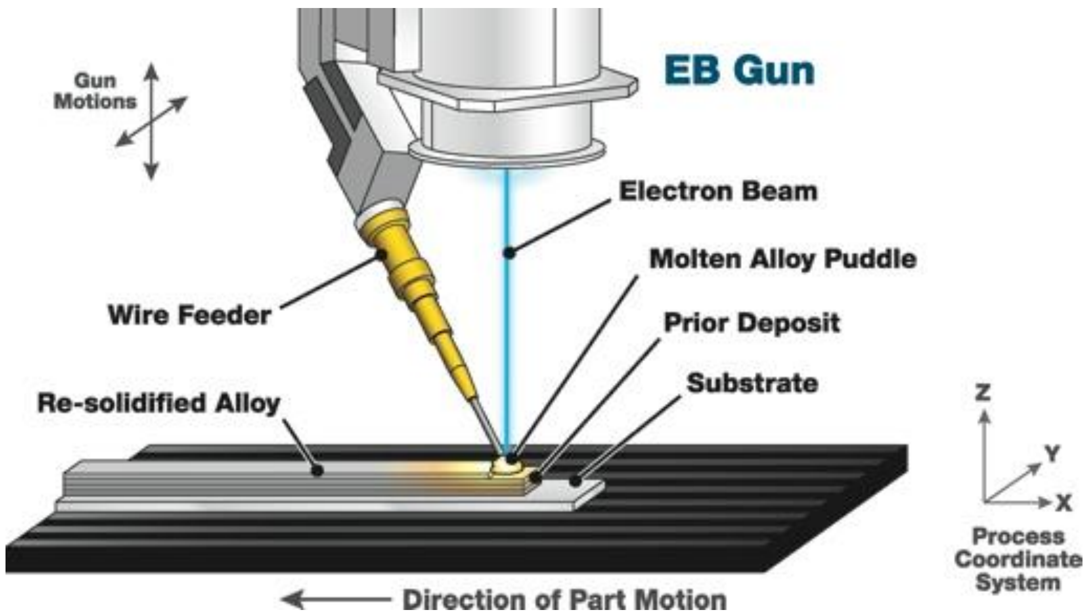
> "Failure is central to engineering. Every single calculation that an engineer makes is a failure calculation. Successful engineering is all about understanding how things break or fail."
> Henry Petroski

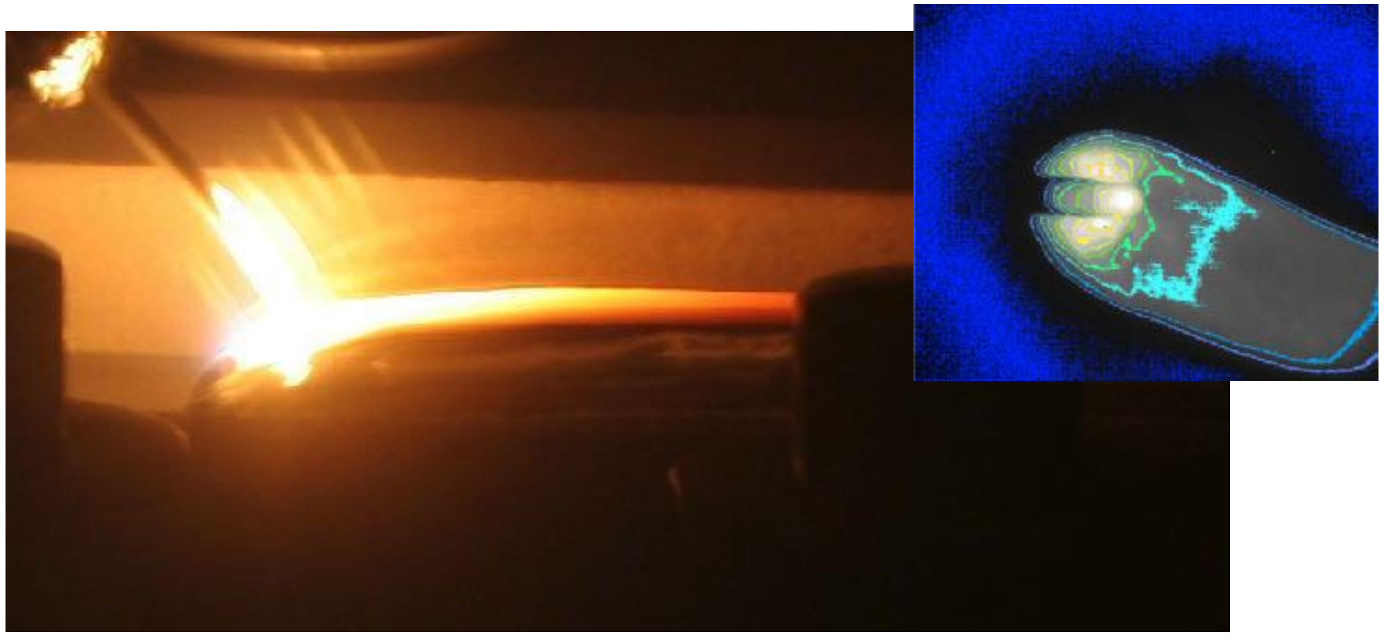
SRAS Basics

Our first data

Possibilities, probabilities, and limitations

Motivation - I (texture in large-scale AM)

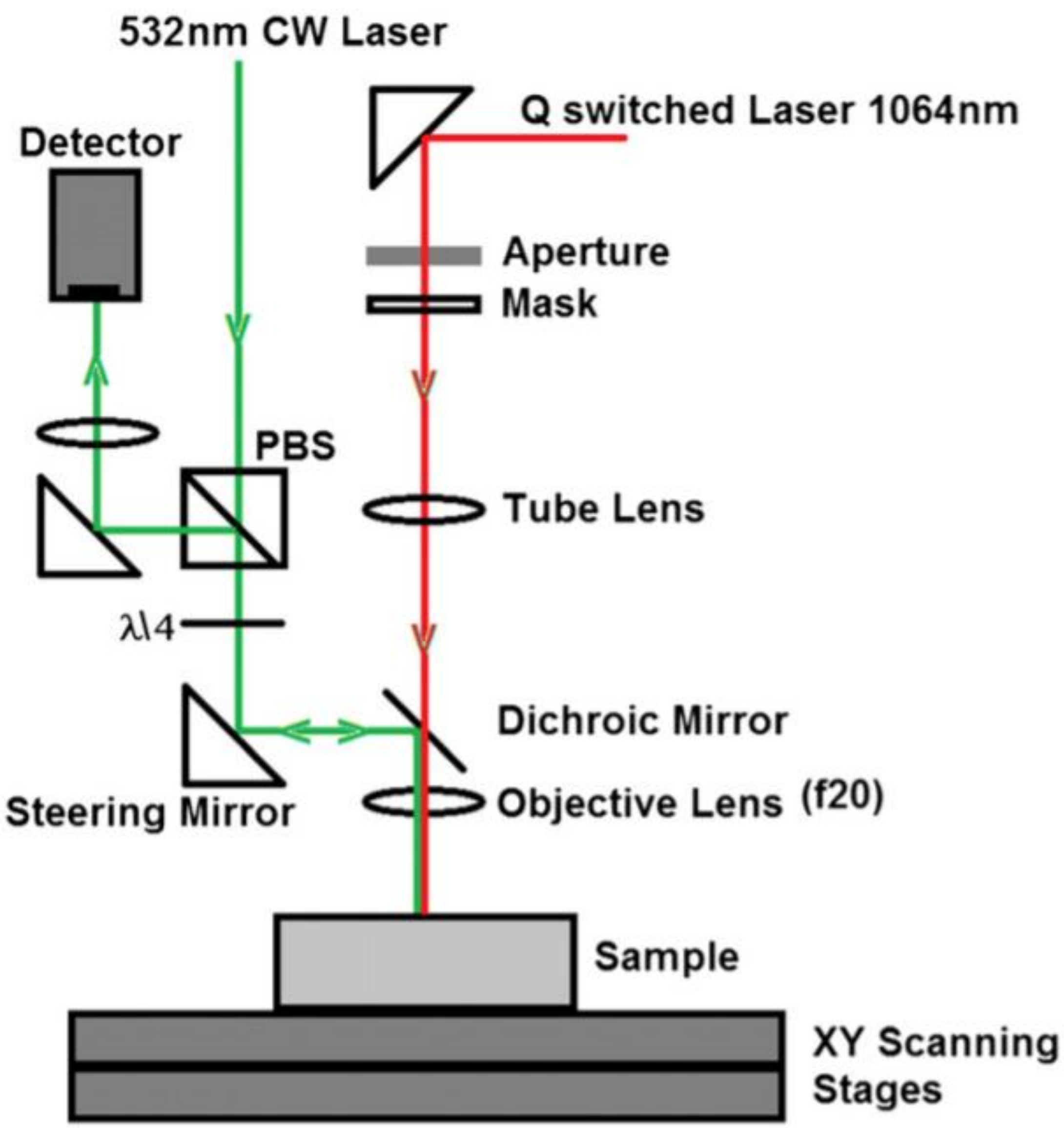




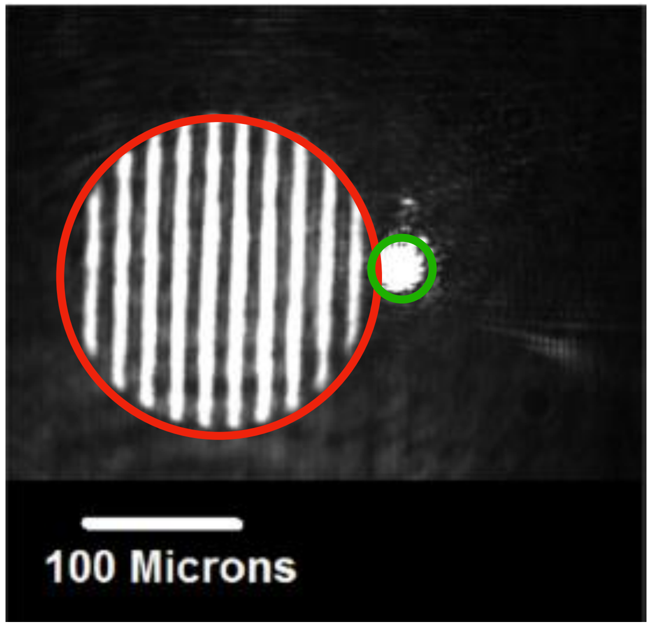
2012 AeroMat presentation: "F-35 Direct Manufacturing: Materia
Qualification Results" June 20, 2012

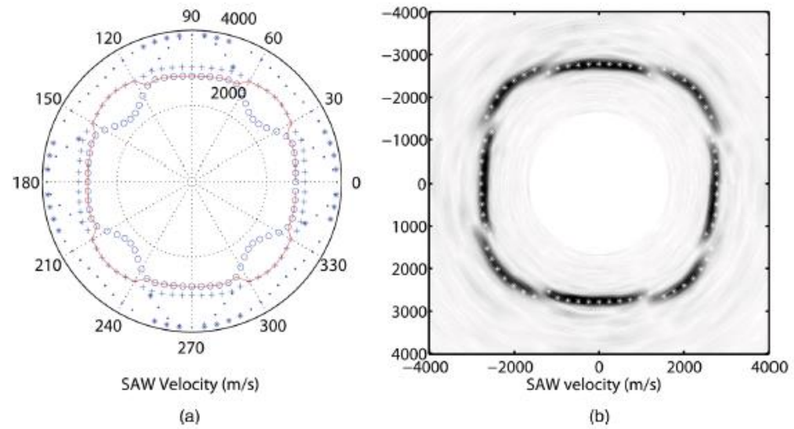
Motivation and overview - II (microtexture?)

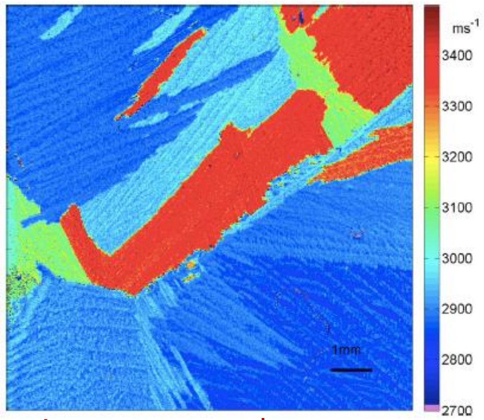
Spatially Resolved Acoustic Spectroscopy



- Laser UT Technique
- Able to detect velocity of SAWs ***(~Mach 9)***
- Can determine crystallographic information through detection of ***nm-level surface displacements***
- coupled with simulation of multiple wave modes (governed by the elastic stiffness tensor, C_{ijkl})
- EBSD-like data with restrictions



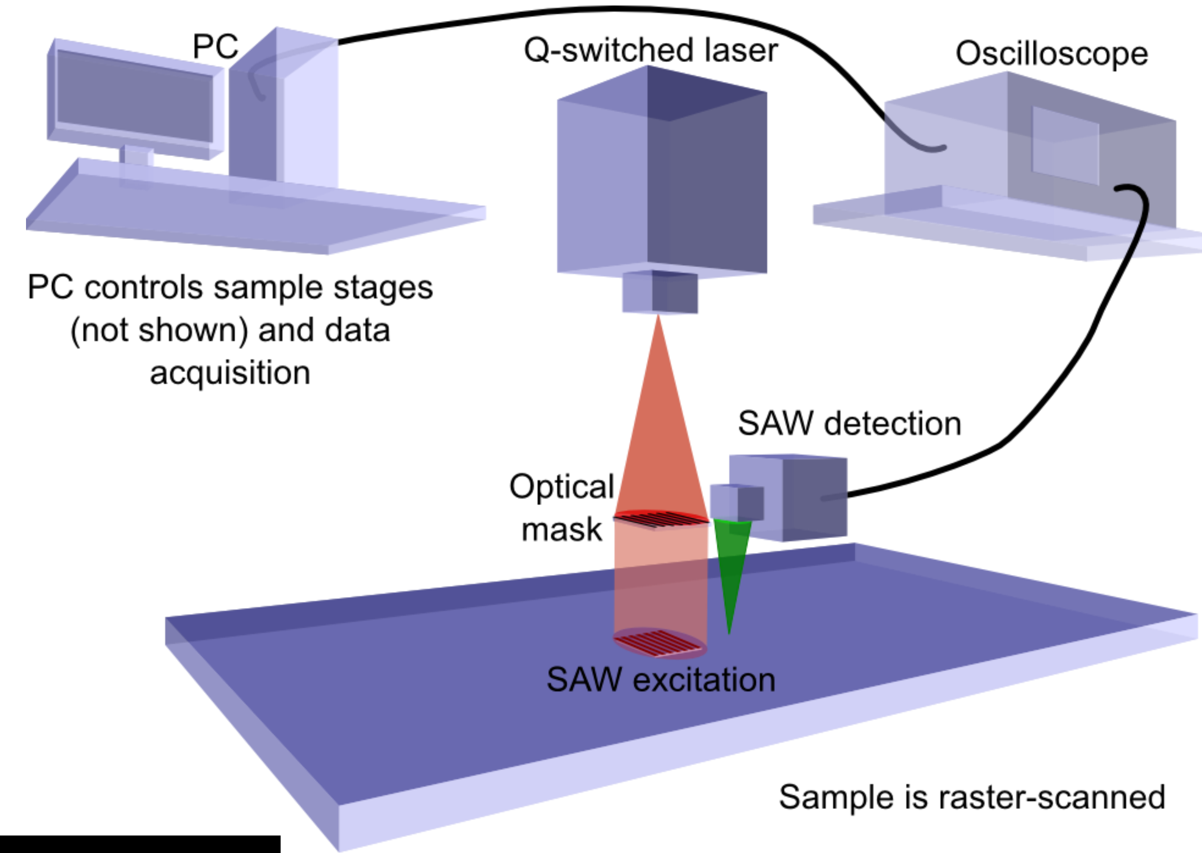


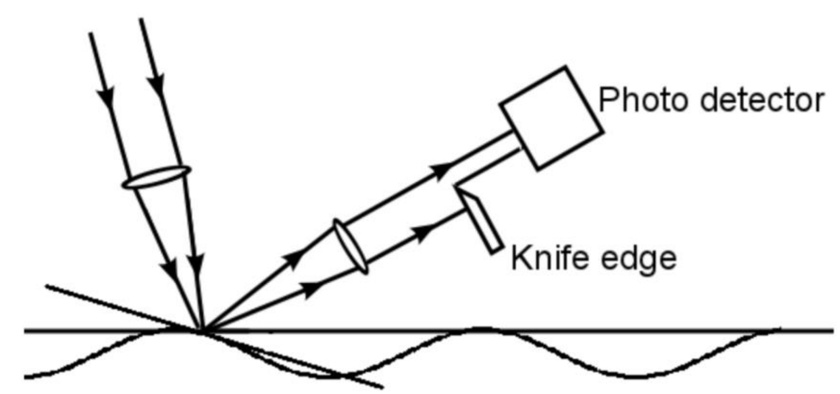


"Spatially resolved acoustic spectroscopy for rapid imaging of material microstructure and grain orientation," Richard J Smith et al, 2014, Meas. Sci. Technol. **25** 055902
DOI: 10.1088/0957-0233/25/5/055902

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
- Calculate the velocity using $v = f\lambda$

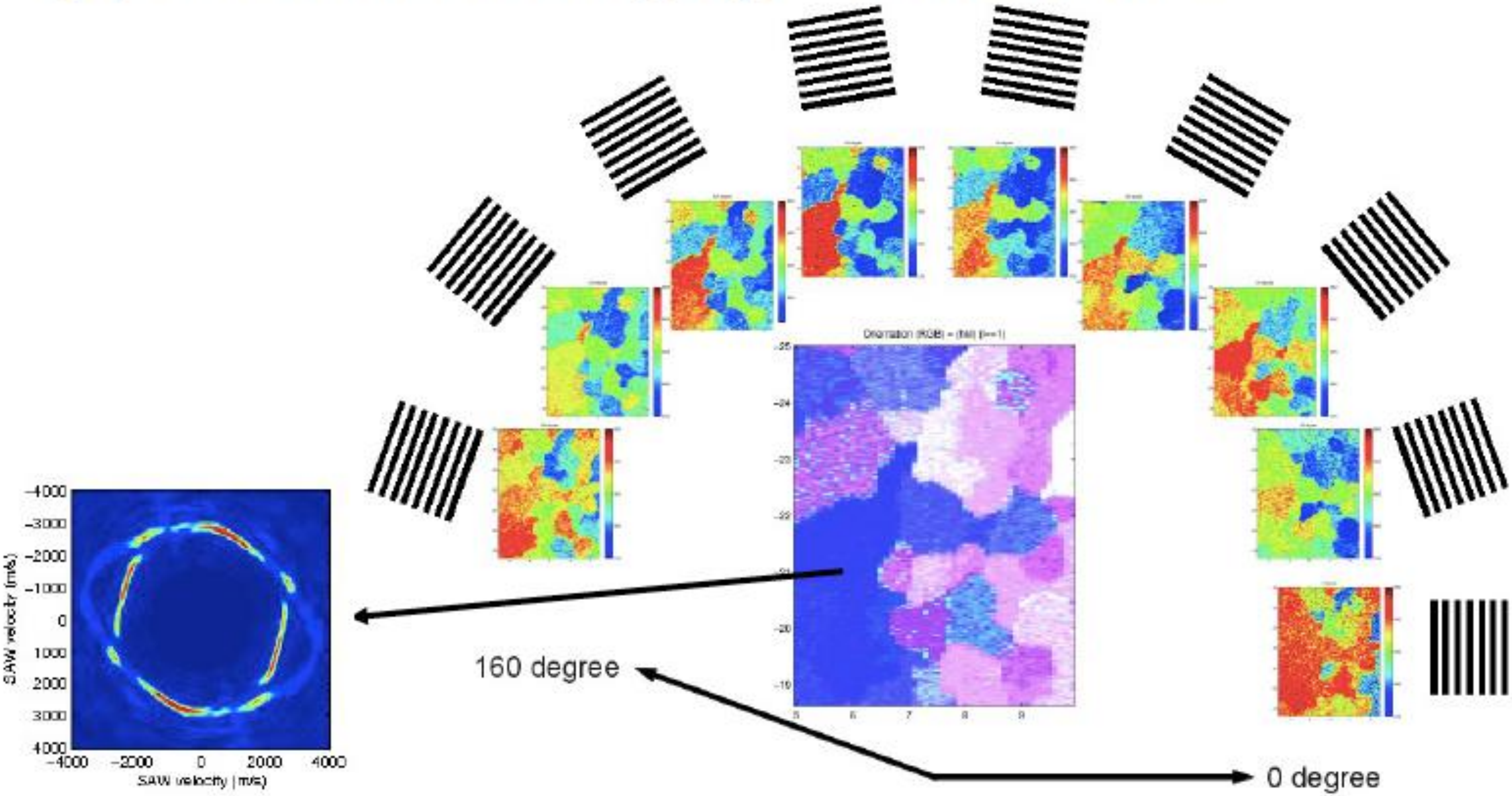




Orientation Determination in practice

University of Nottingham
UK | CHINA | MALAYSIA

(2) Orientation imaging – Collect data

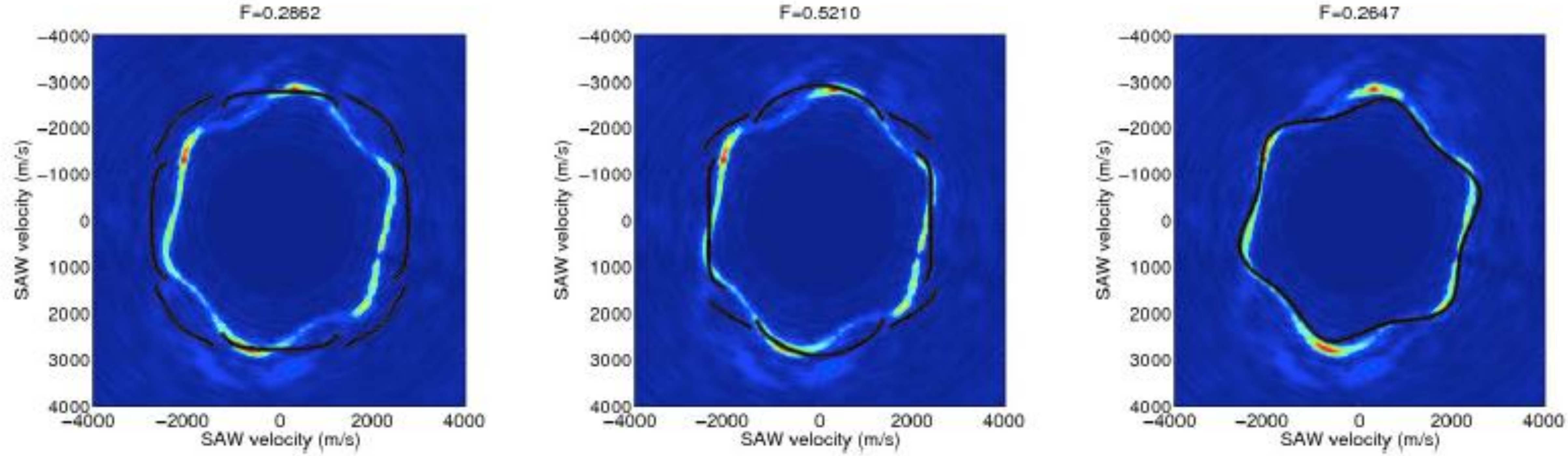


Orientation Determination in practice

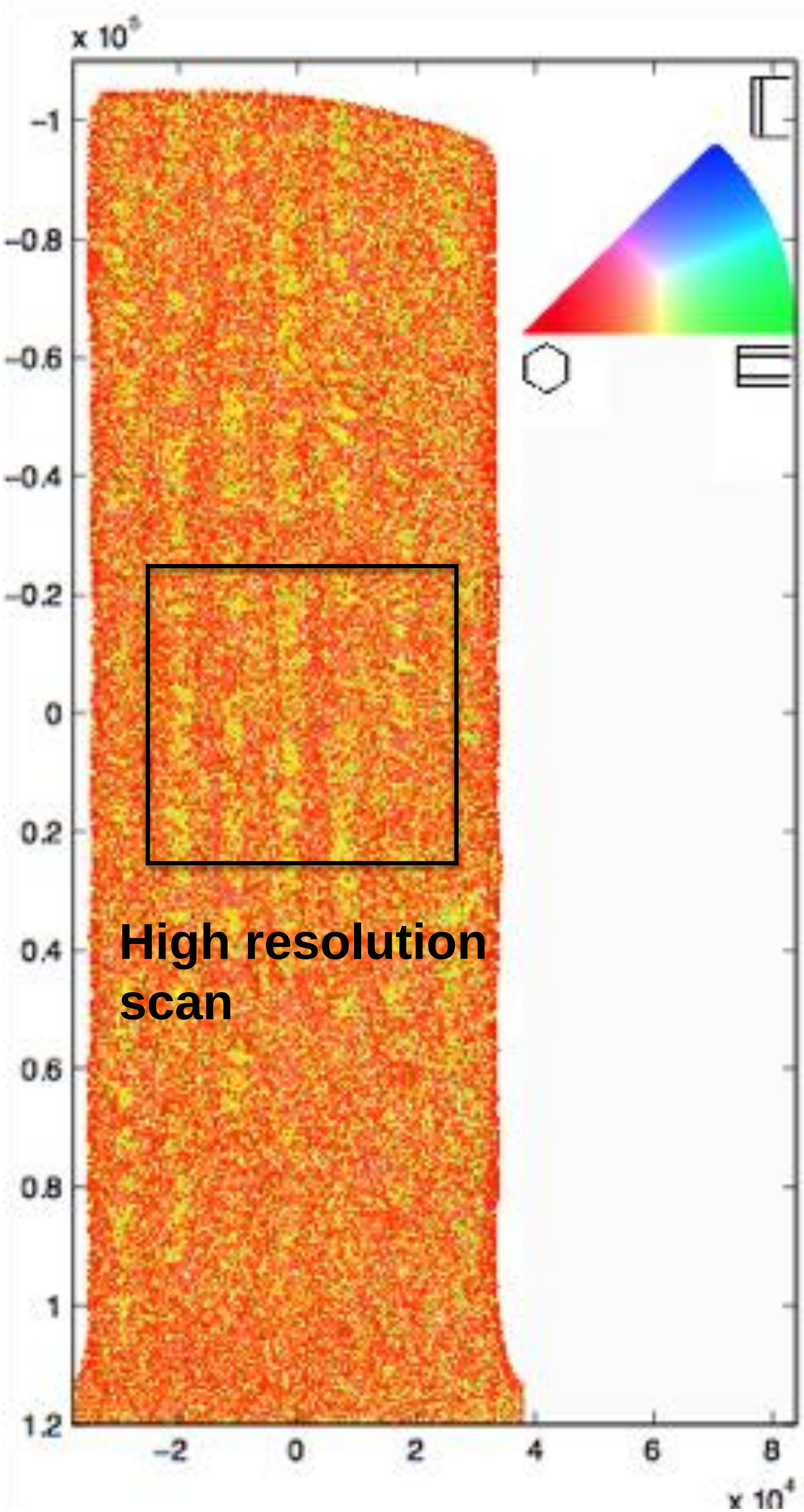
University of Nottingham
UK | CHINA | MALAYSIA

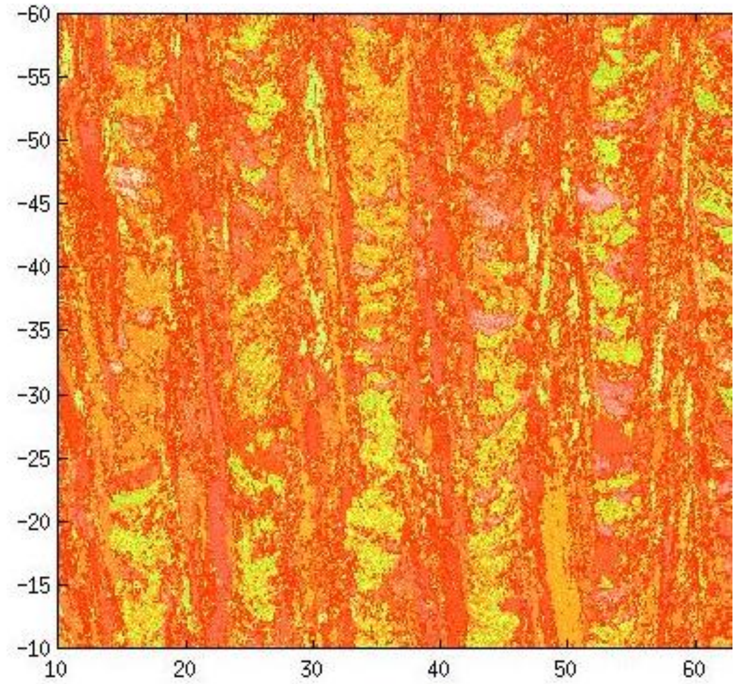
(3) Orientation imaging - fit to data

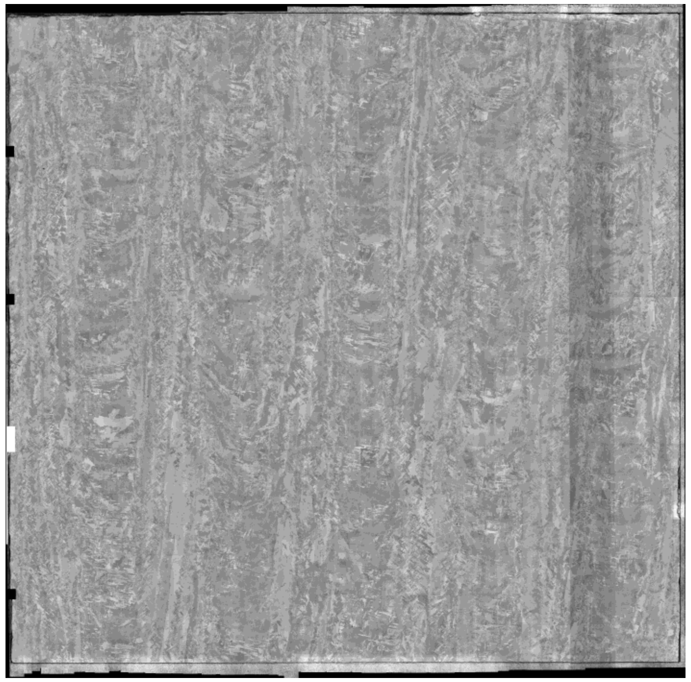
$$F(h,k,l,\theta) = \sum_{\phi=0}^{\phi=\frac{n-1}{n}\pi} A(\phi, v_{(h,k,l,(\phi-\theta))}), \; (n = 1,\ 2,\ 3,\ \ldots)$$

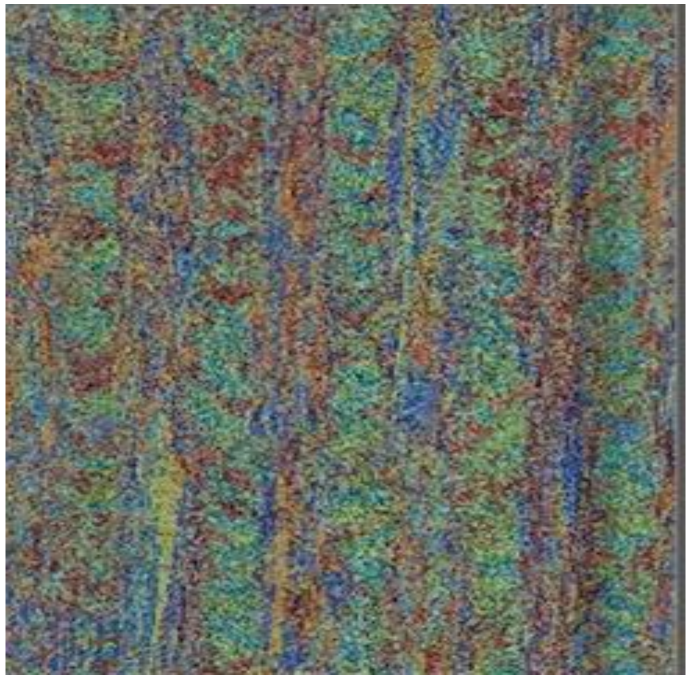


- The merit function is simply the sum of the amplitude under the black asterisks on the graph
- Repeat this procedure for all the combinations of plane and propagation direction

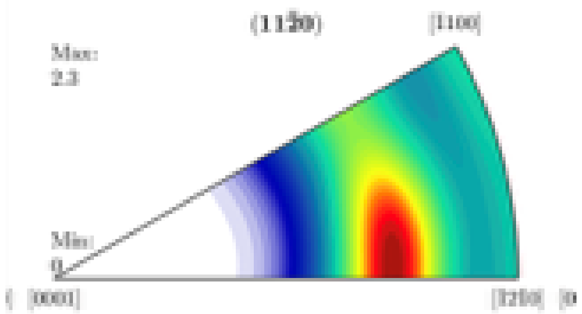

x 10^5
-1
-0.8
-0.6
-0.4
-0.2
0
0.2
0.4
0.6
0.8
1
1.2
-2
0
2
4
6
8
x 10^4
High resolution scan

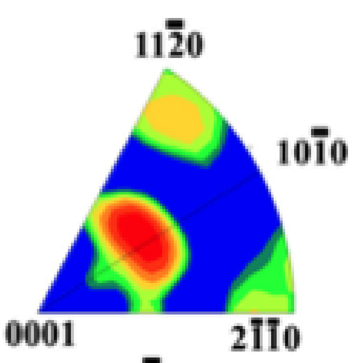

-60
-55
-50
-45
-40
-35
-30
-25
-20
-15
-10
10
20
30
40
50
60

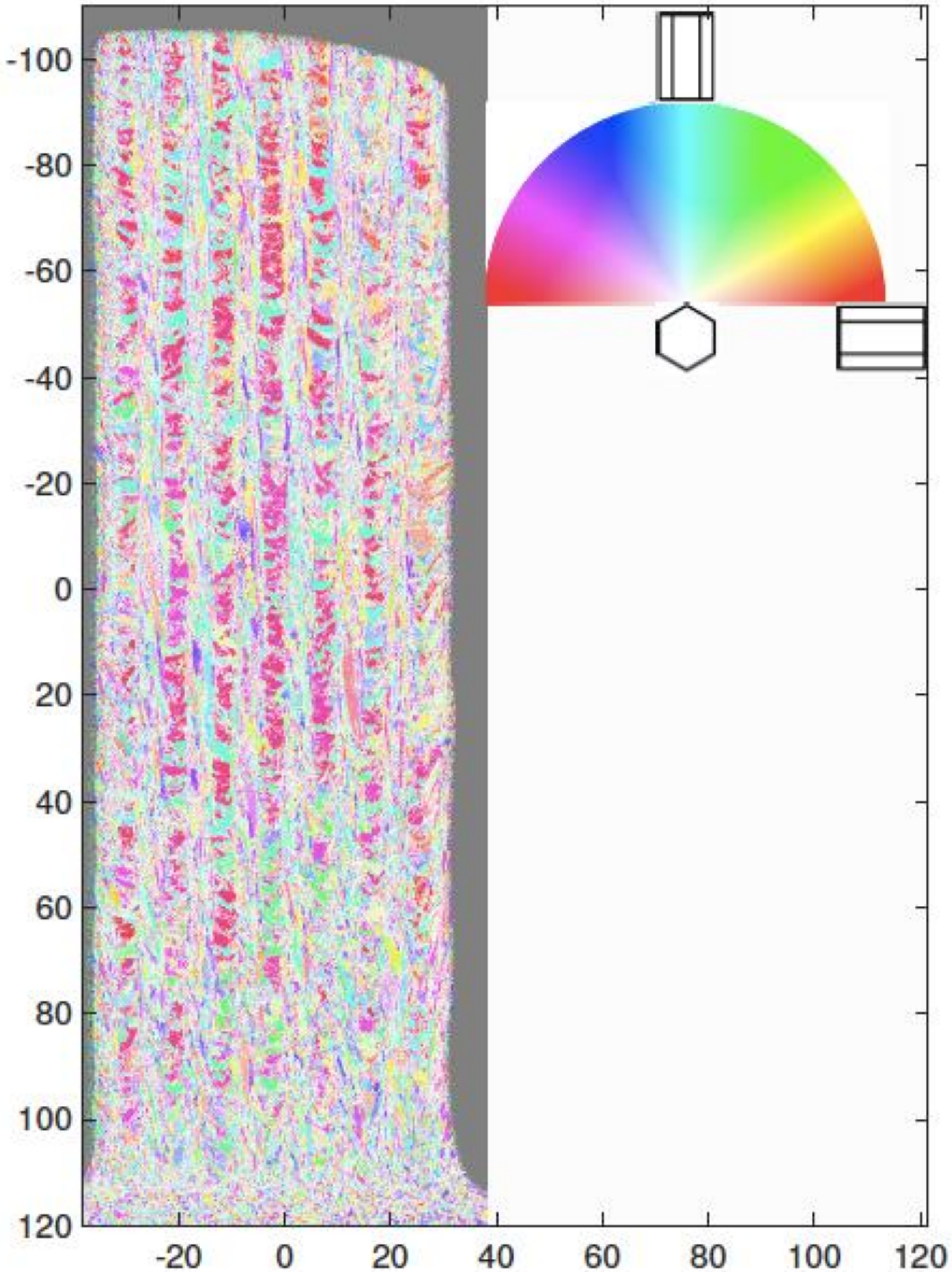


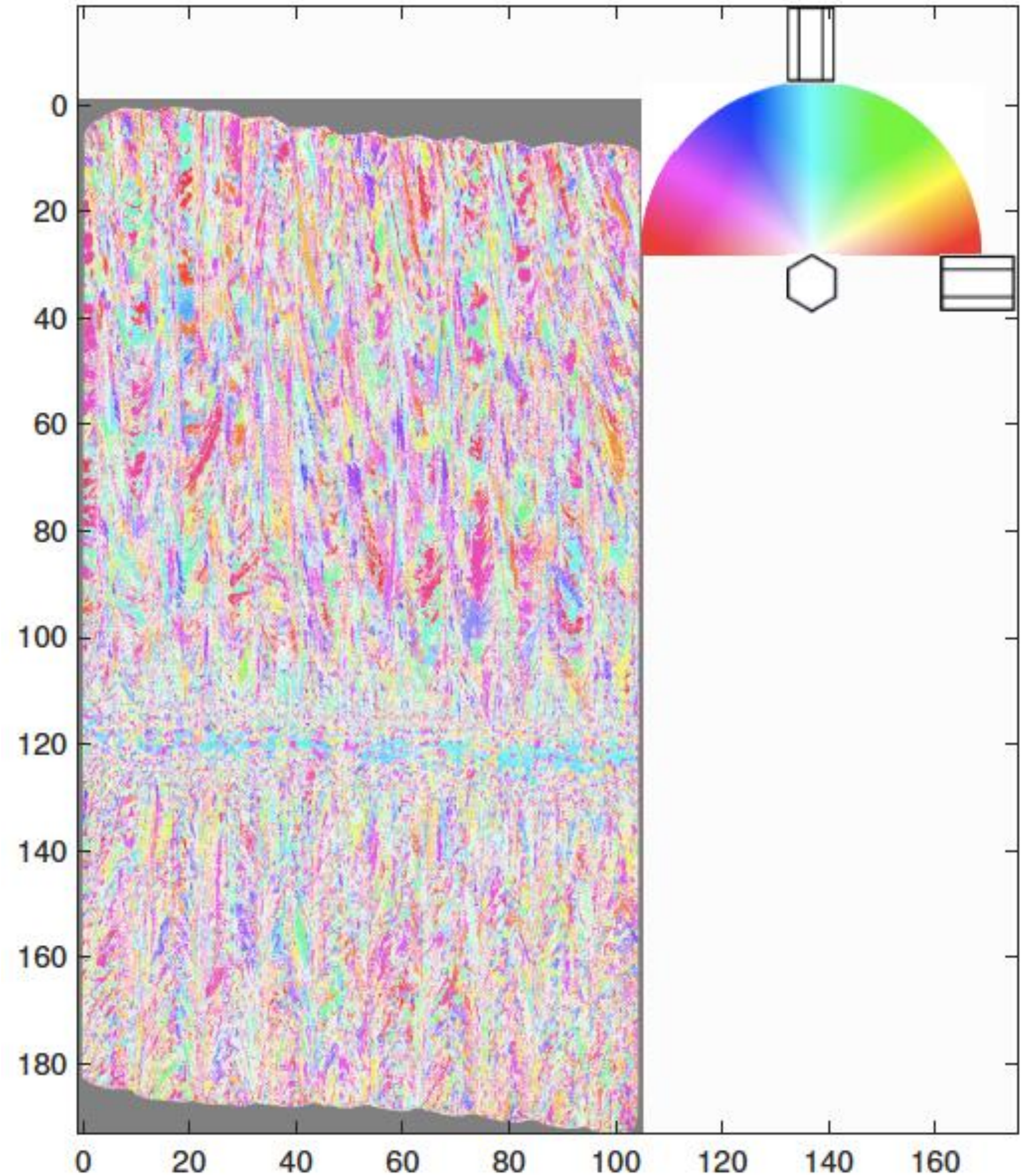



5
1
6
2
7
3
8
4
9


Max:
2.3
Min:
0


11$\bar{2}$0
10$\bar{1}$0
0001
2$\bar{1}\bar{1}$0





In these two scans, we *may* have more orientation data than all EBSD scans of AM Ti-6Al-4V combined.

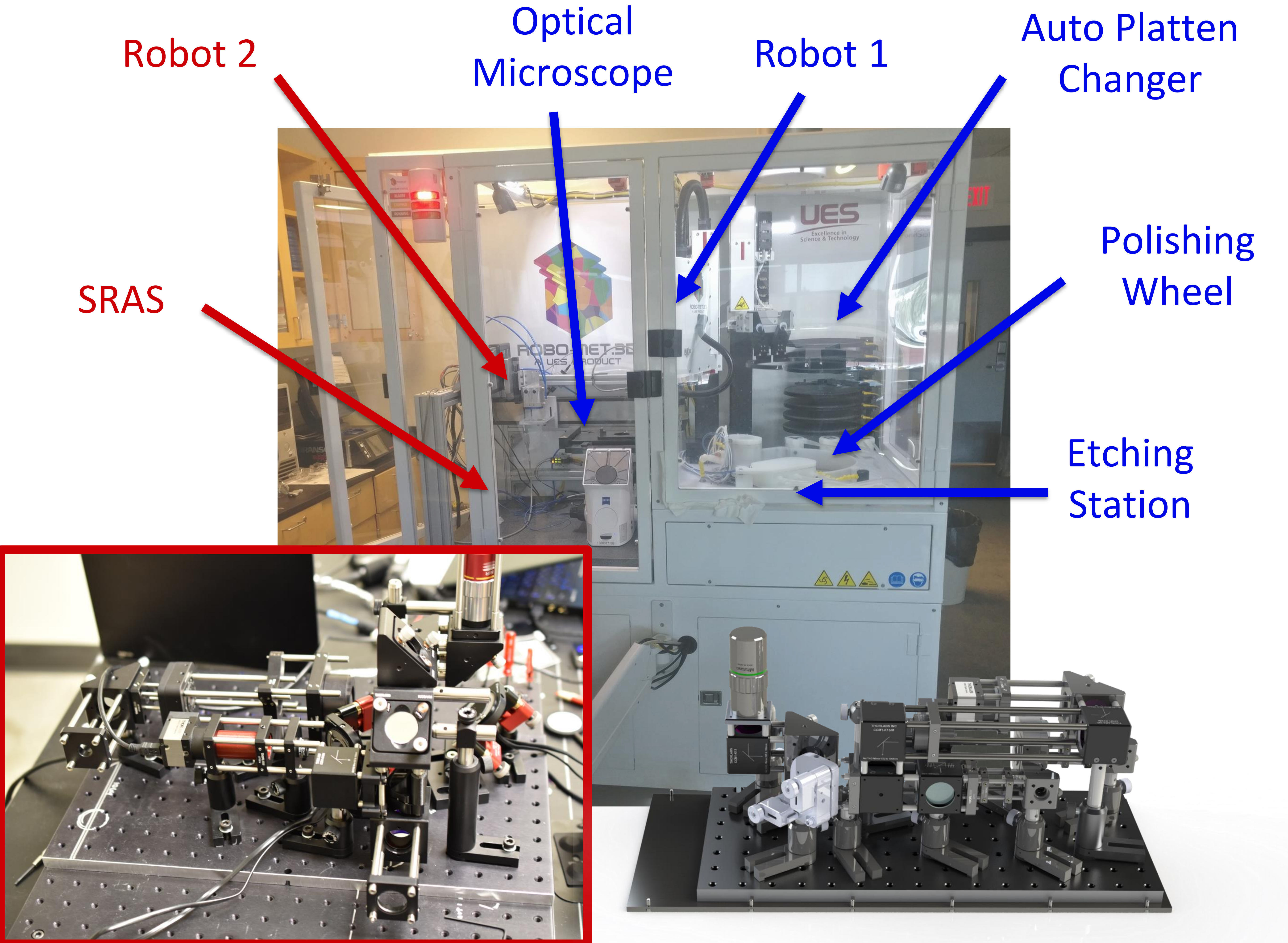
So, let's make a SRAS system…

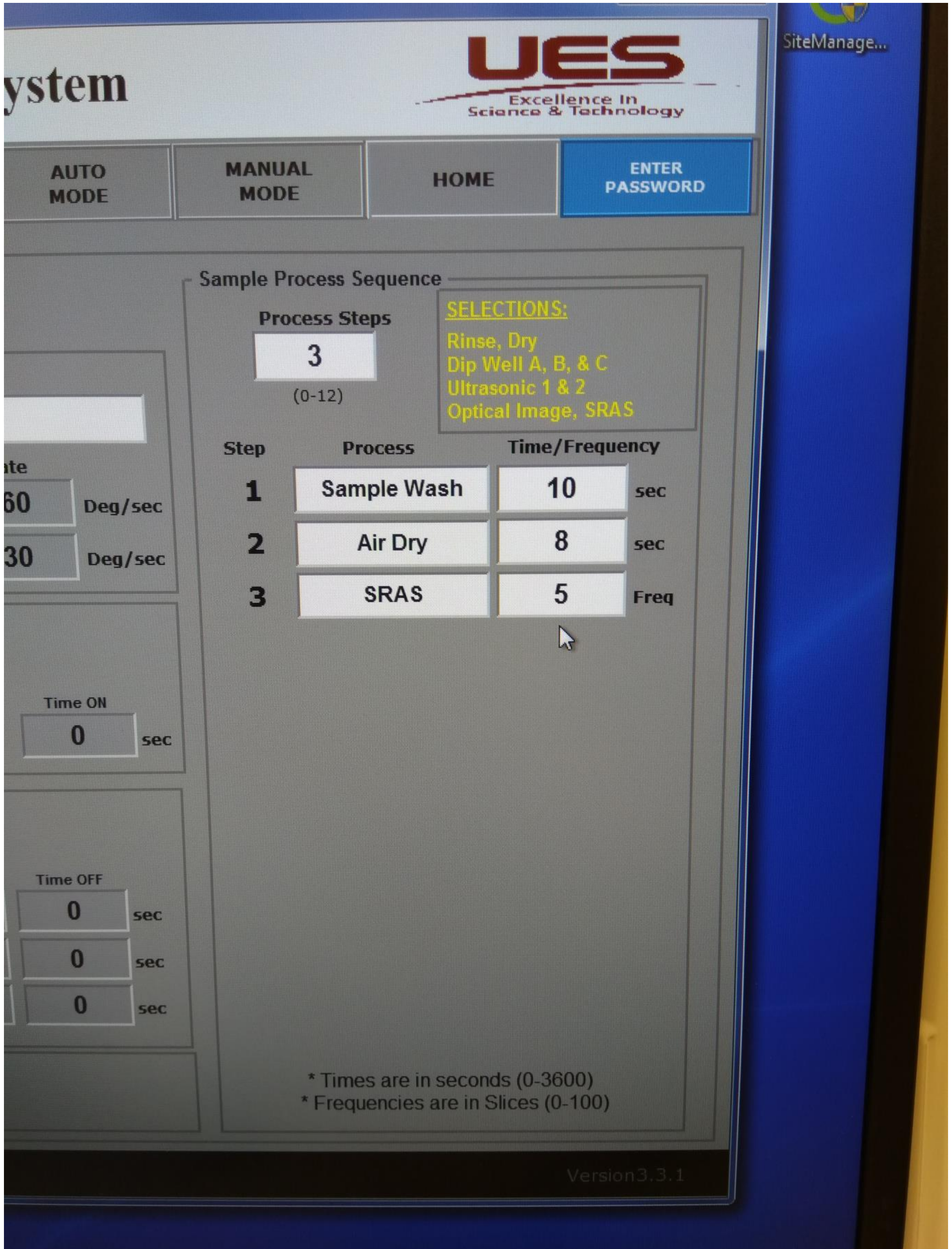
…but let's integrate it into a serial sectioning tool

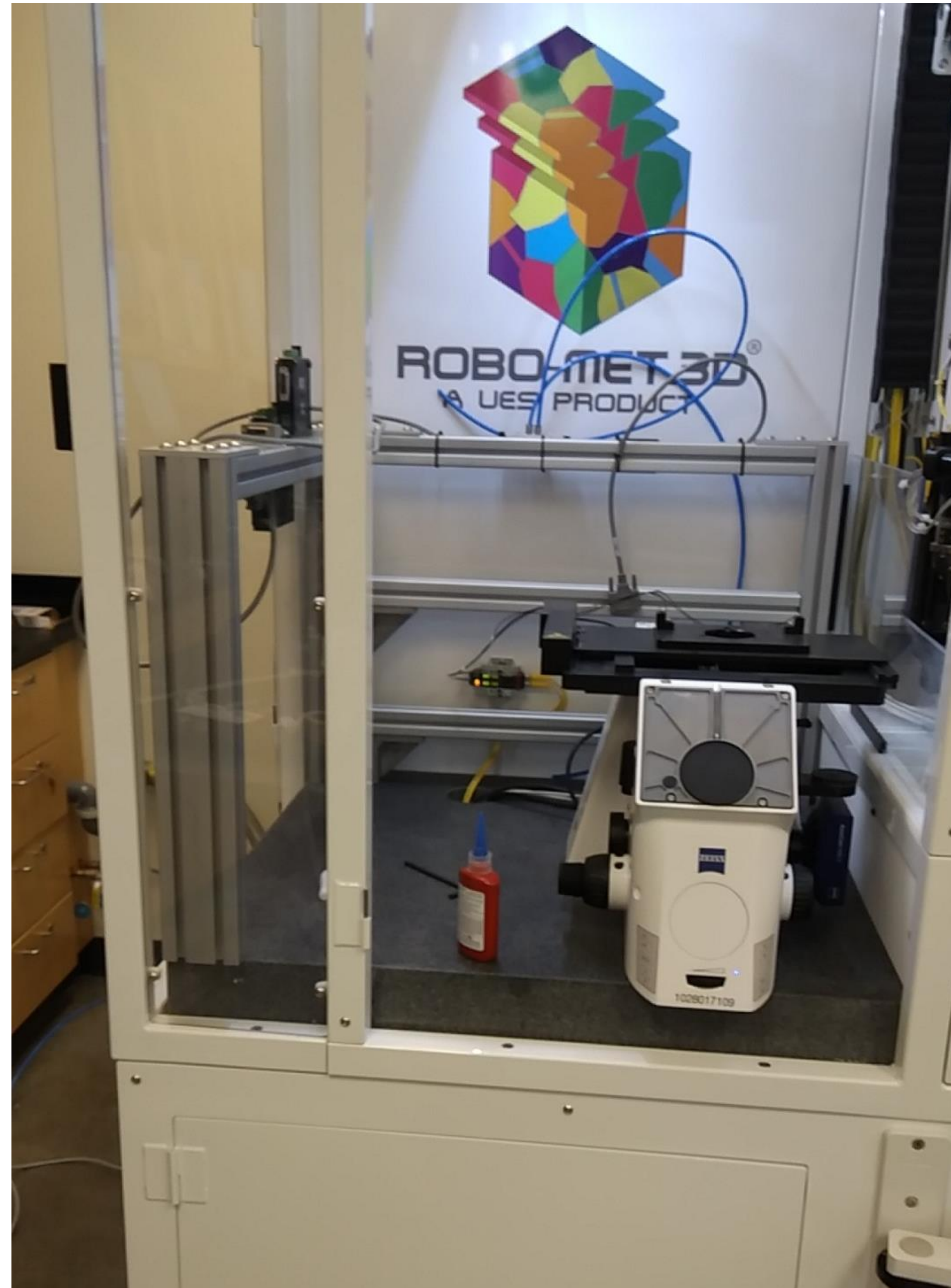
(as if SRAS is not hard enough)

With credit given to:

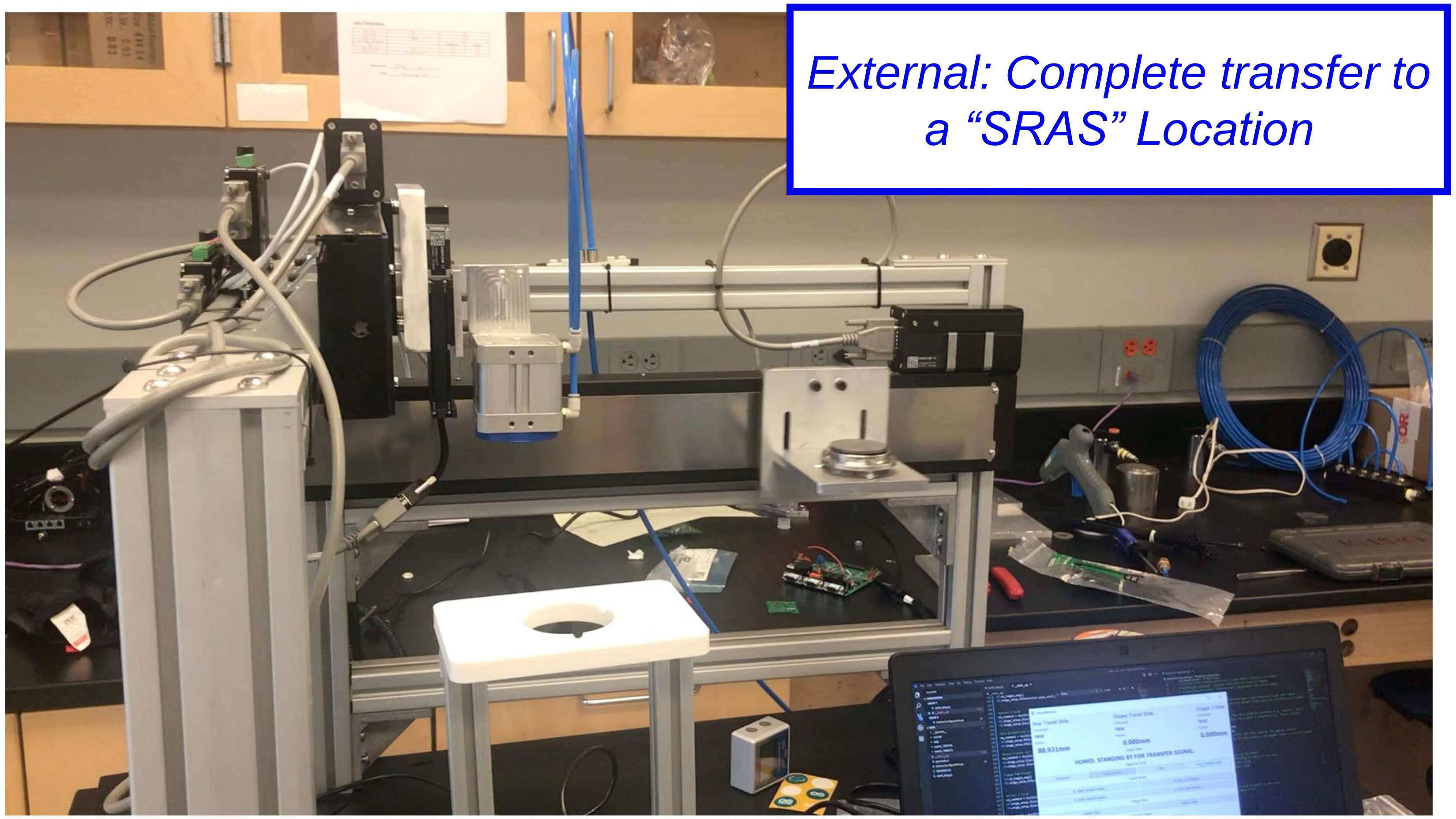
- **Thomas Ales**
- **The Art of Electronics (Horowitz & Hill)**


Robot 2
Optical
Microscope
Robot 1
Auto Platten
Changer
Polishing
Wheel
SRAS
Etching
Station
UES
ROBO-MET.3D


ystem
UES
Excellence In
Science & Technology
SiteManage...
AUTO MODE
MANUAL MODE
HOME
ENTER PASSWORD
Sample Process Sequence
Process Steps
3
(0-12)
SELECTIONS:
Rinse, Dry
Dip Well A, B, & C
Ultrasonic 1 & 2
Optical Image, SRAS
Step
Process
Time/Frequency
1 Sample Wash 10 sec
2 Air Dry 8 sec
3 SRAS 5 Freq
60 Deg/sec
30 Deg/sec
Time ON
0 sec
Time OFF
0 sec
0 sec
0 sec
* Times are in seconds (0-3600)
* Frequencies are in Slices (0-100)
Version3.3.1

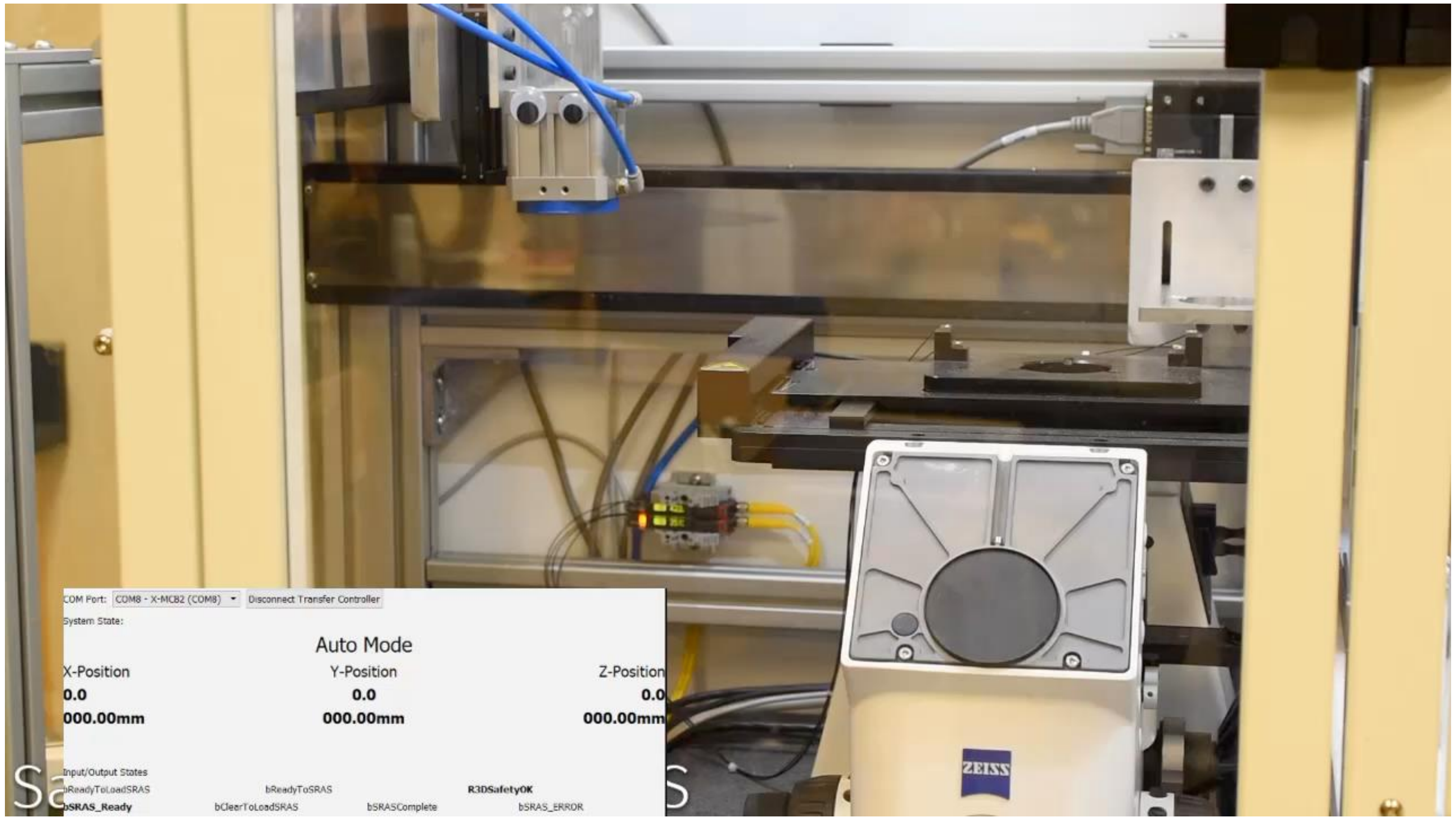

ROBO-MET.3D
A UES PRODUCT

Automated robot transfer system



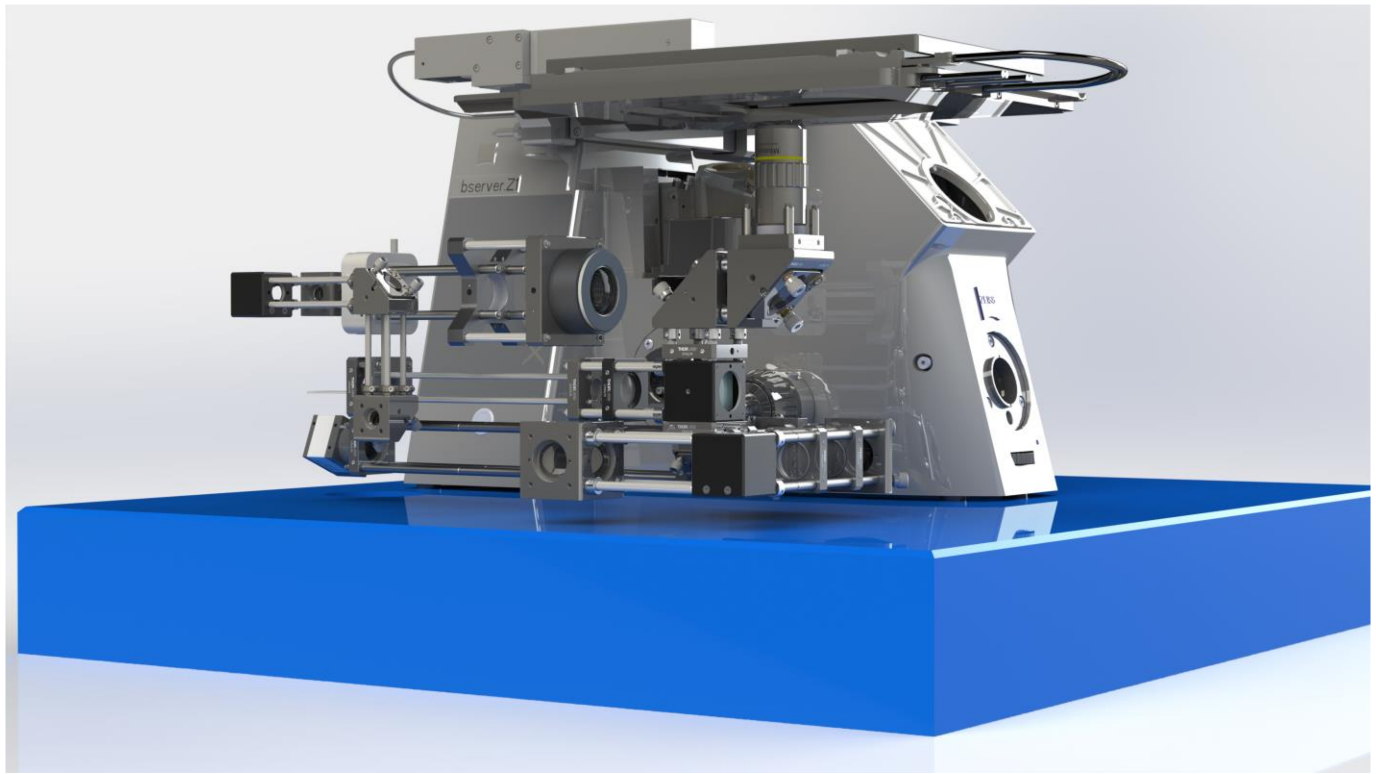
External: Complete transfer to a “SRAS” Location

Below: Internal (no drop-off) with code. Precision of location.

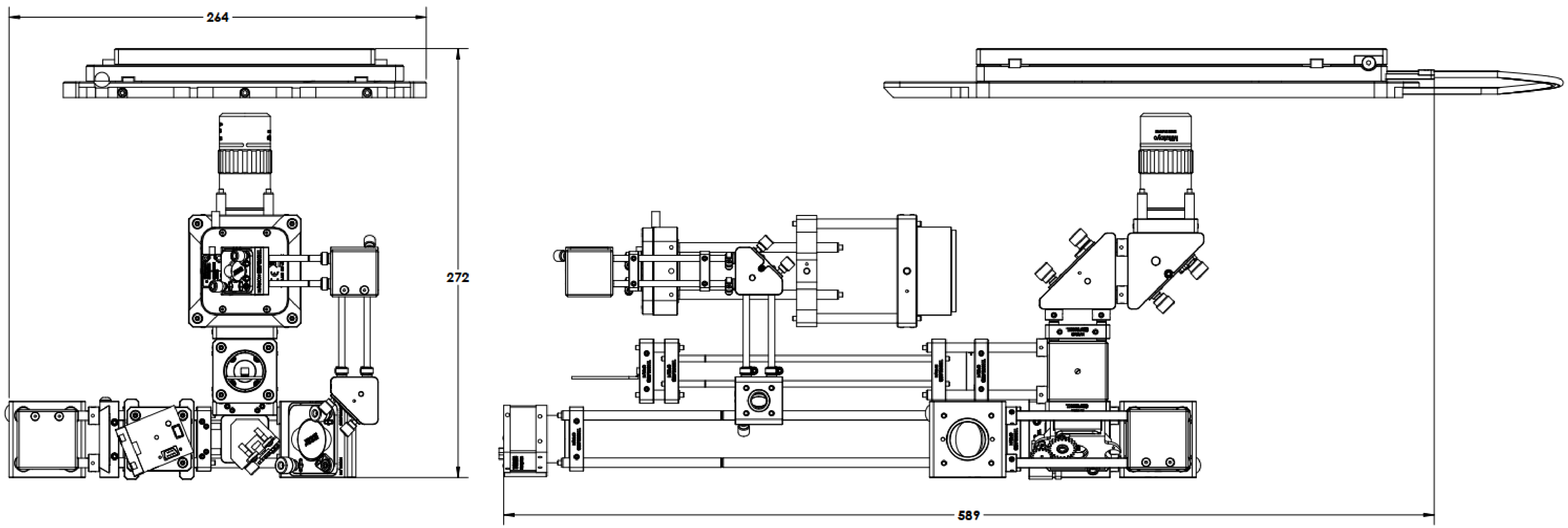


Possibility: Expansion for other “drop off” sites. Ultrasound? X-Ray? SEM?

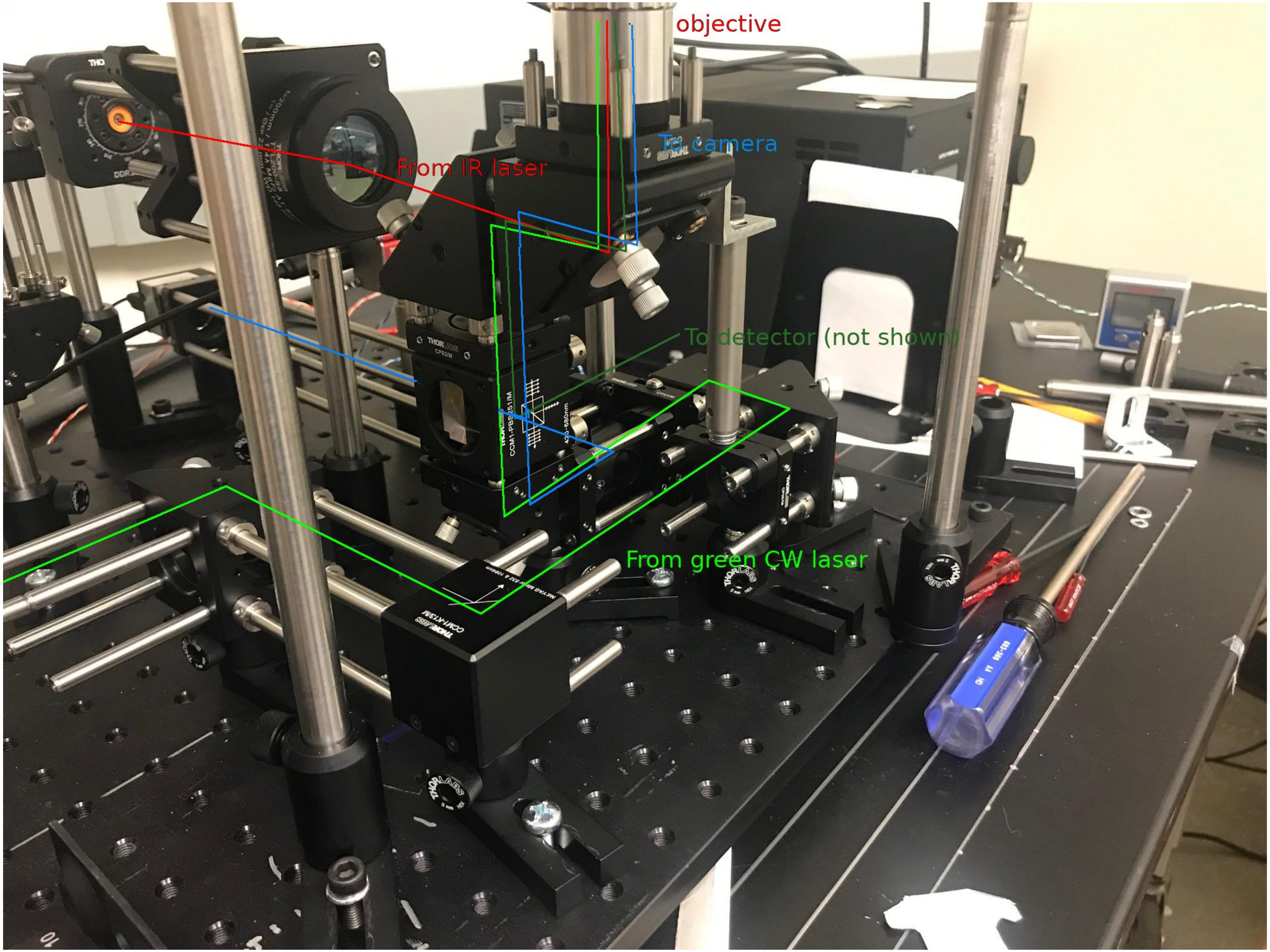
Engineering a new system

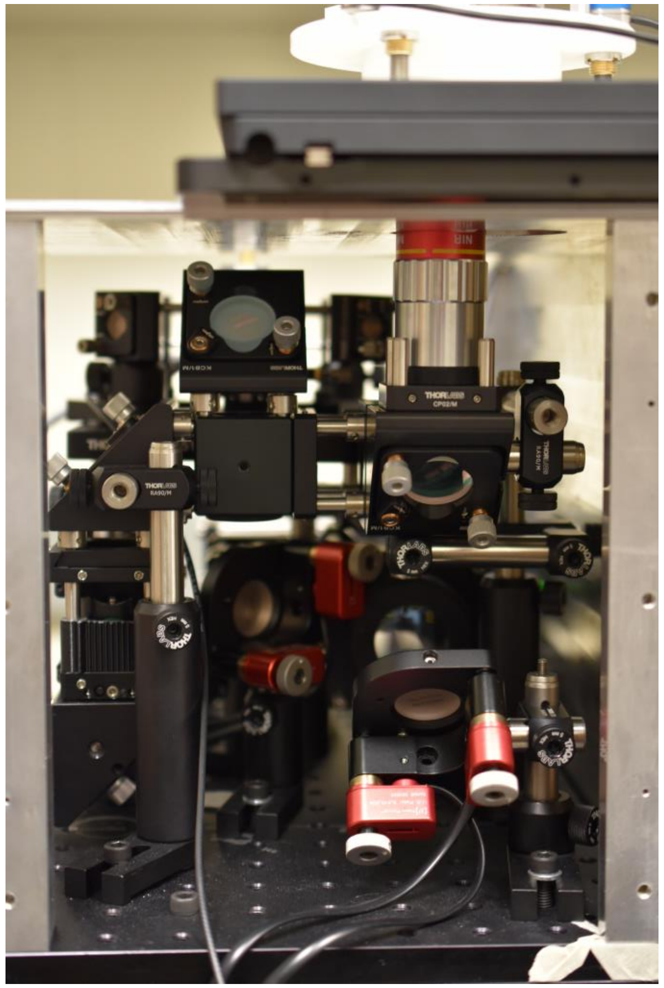


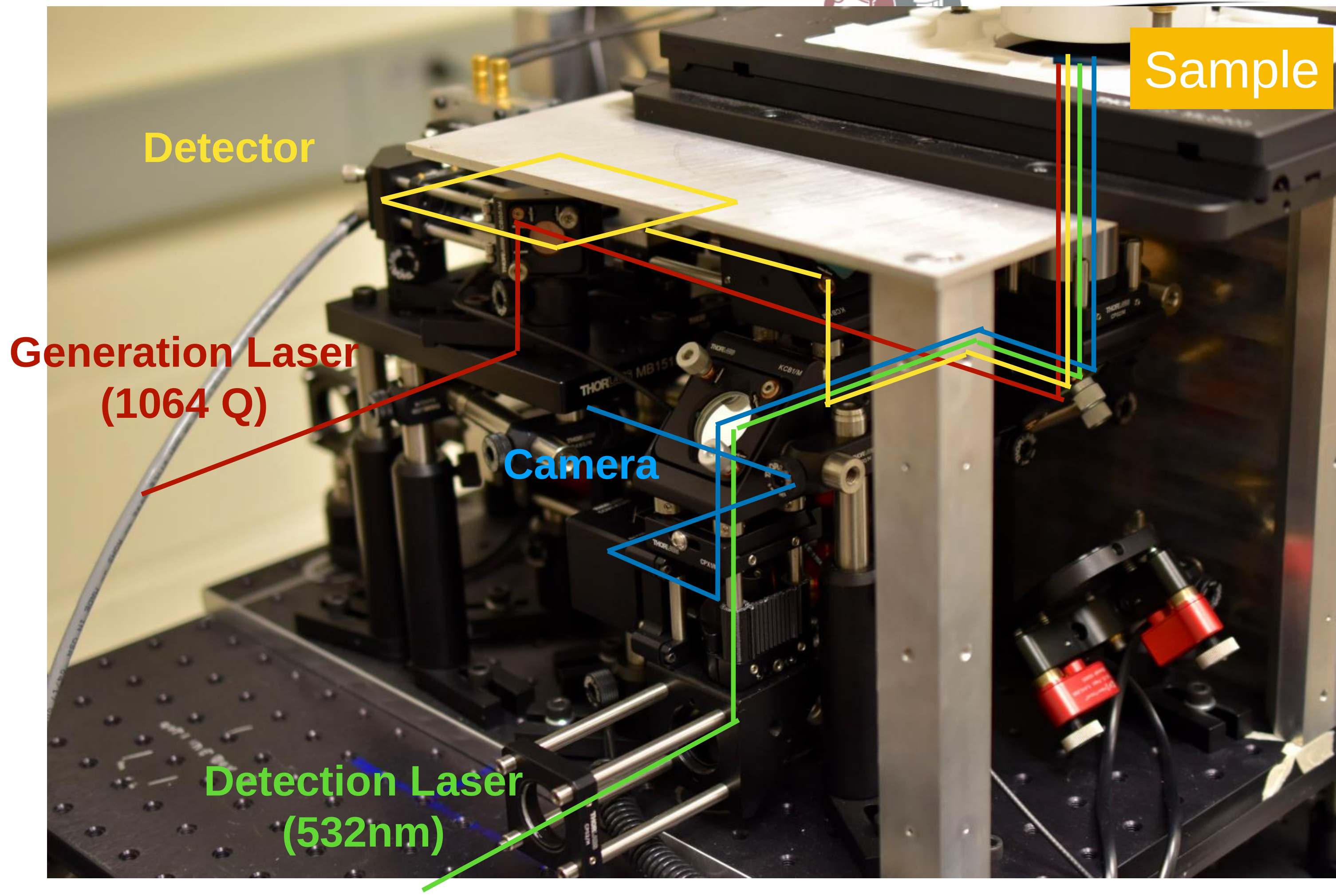
- Must wrap a 2D system to a 3D package
- Highly space limited
- Safety!
- Detector components no longer exist
- Bandwidth (150-500 MB/s if we collected everything!)
- Thus, we need triggers
- Saves on data (makes TBs datasets GB instead - RAW)
- A true engineering challenge!!



First Original 3D Beam Path





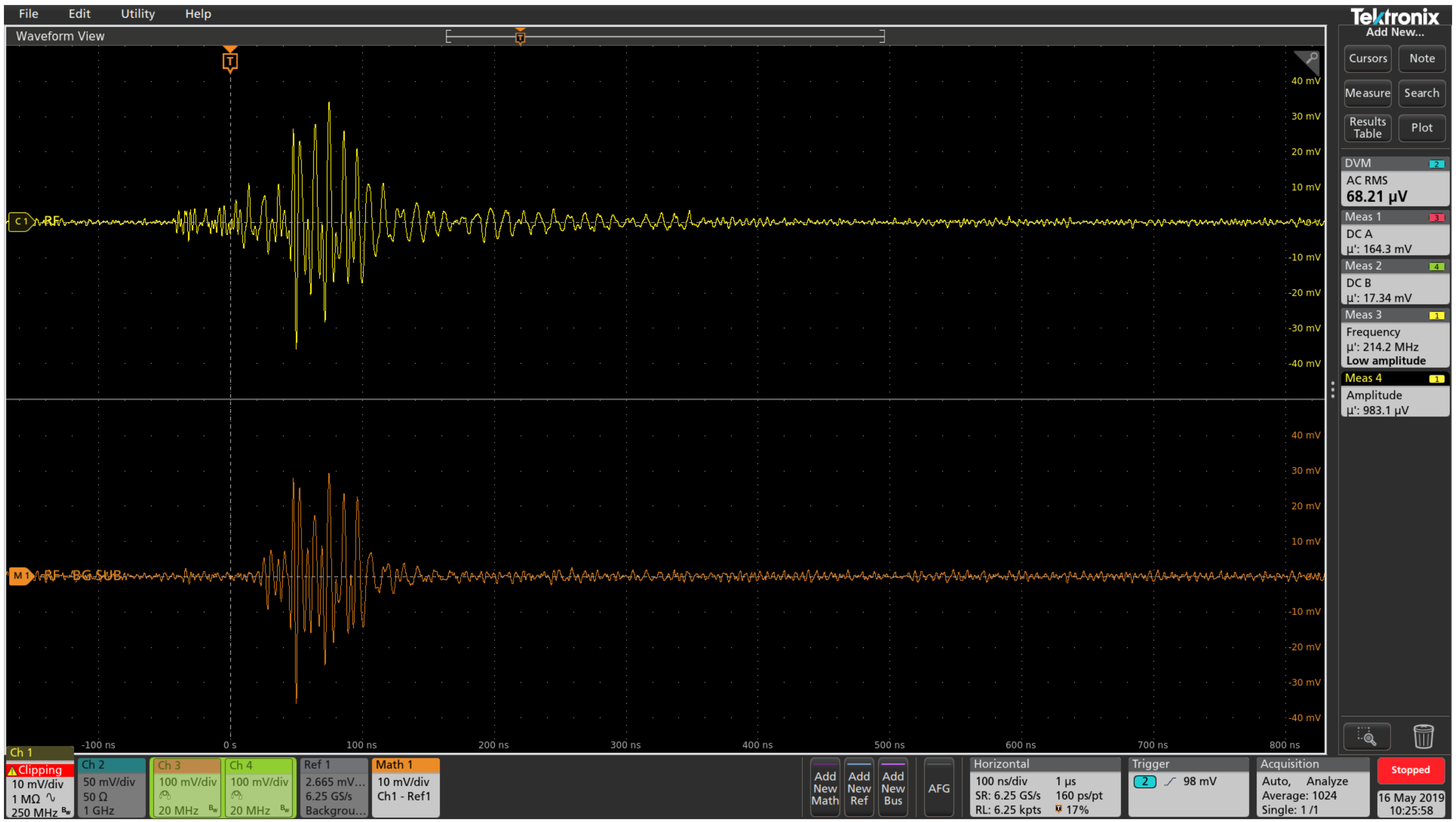


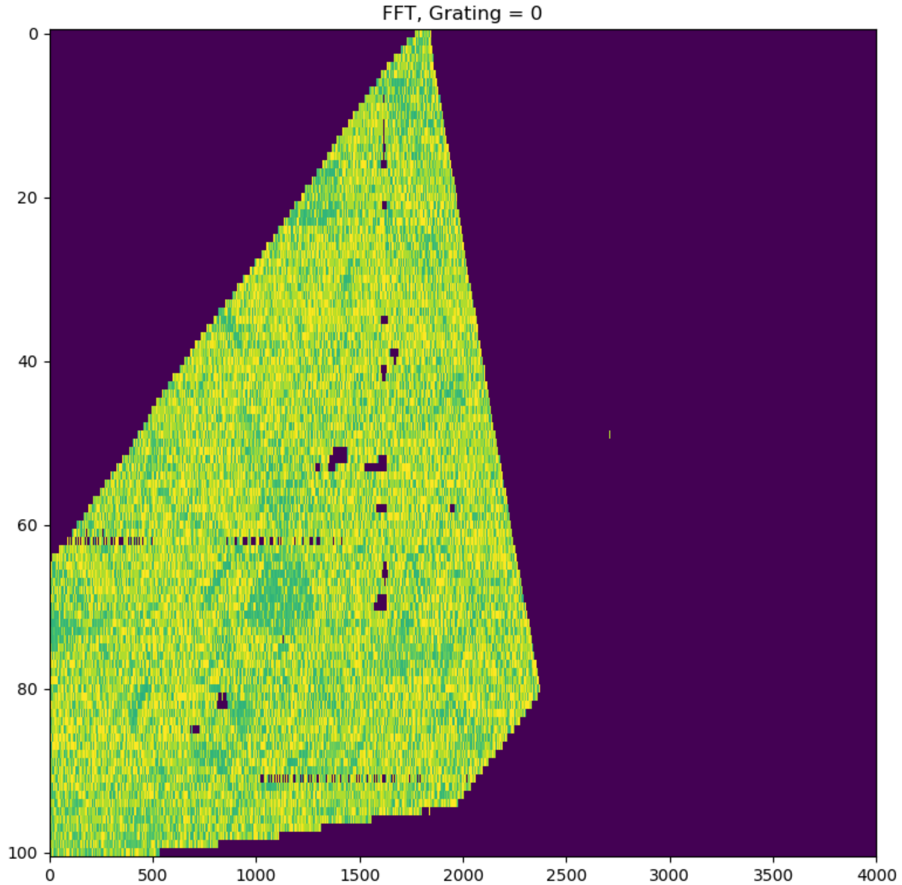
Subsystem		
Generation Laser	Type	Q-switched
	Wavelength	1064 nm
	Pulse Energy	>50 uJ
	Pulse Duration	<900 fs
	Frequency	200-100 kHz
Detection Laser	Type	Continuous
	Wavelength	532 nm
	Mode	TEM00
	Power	0-500 mW ~200 mW

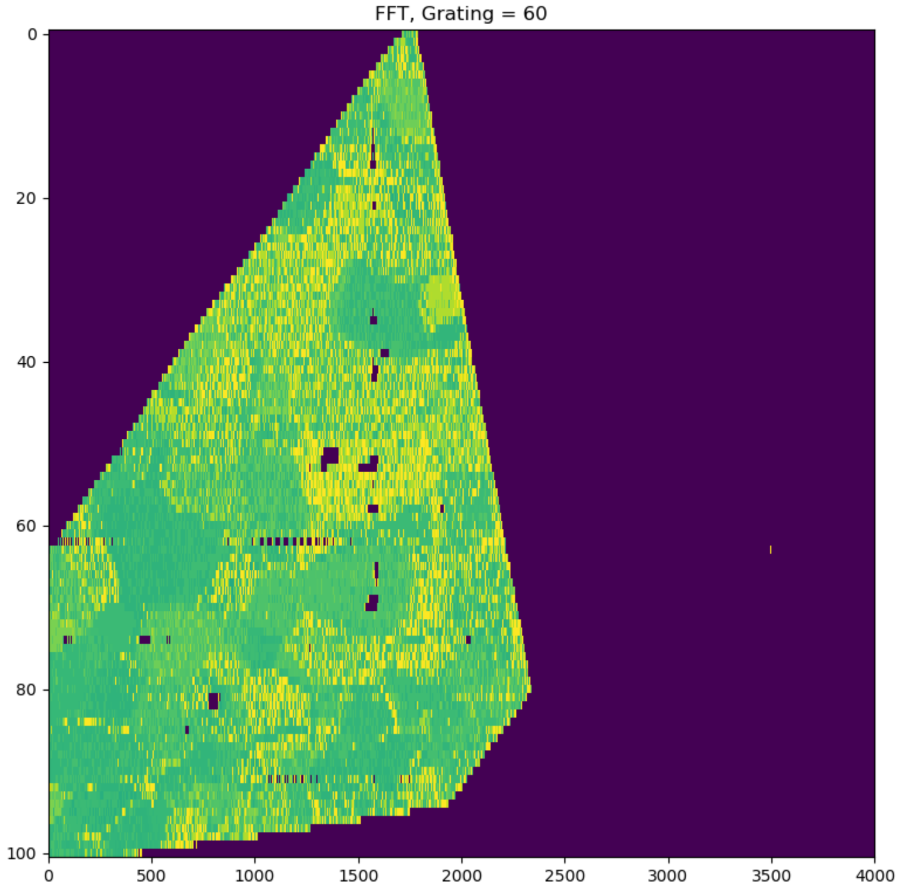
Subsystem		
Stage	Speed	250 mm/s
	Acceleration	2500 mm^2/s
	Backlash	None
	Accuracy	<0.25 um
	Incremental Movement	<100 nm
Detector	Type	Balanced Split PD
	Generation	New (1st since ~2011)
	Frequency	< 500 MHz
	Spatial Resolution	~ 25 um

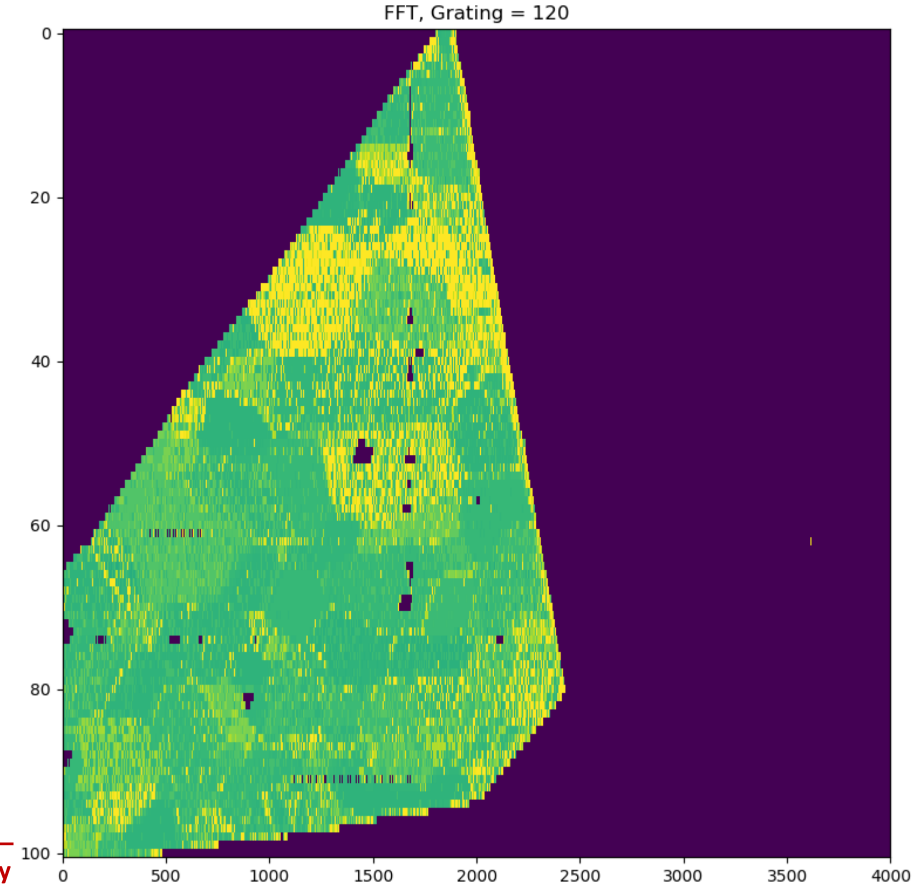
Detector (Nottingham "Slow")

First signals (June)

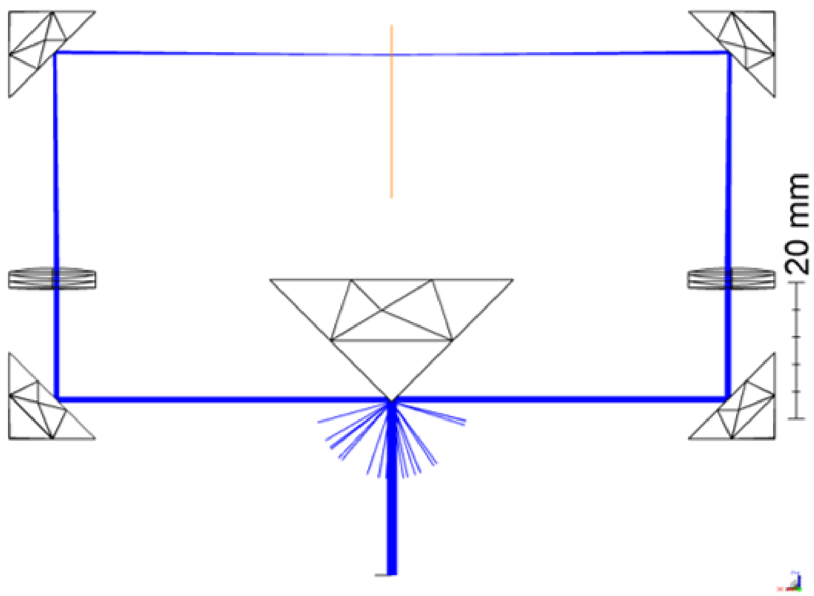


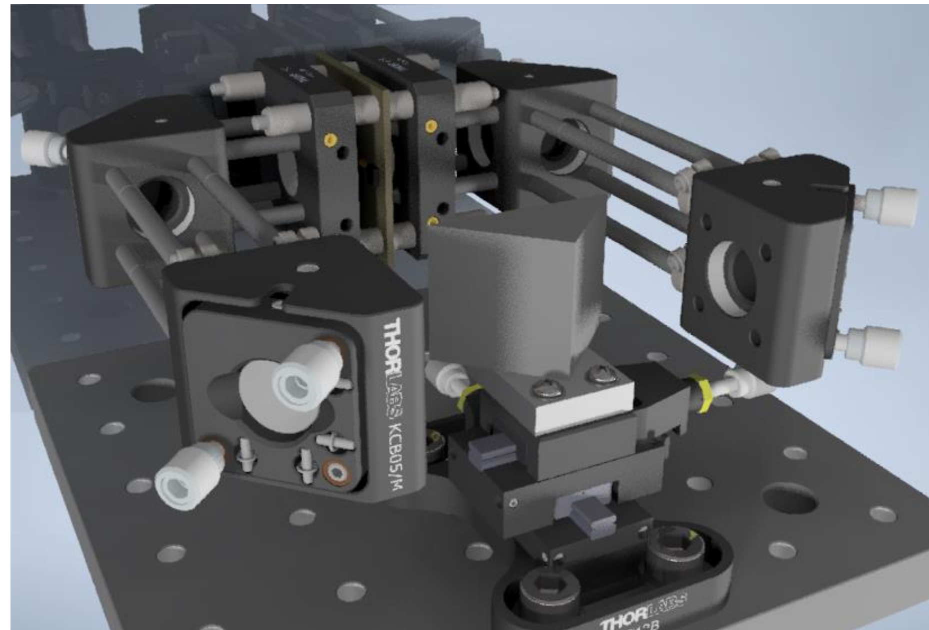

FFT, Grating = 0
0
20
40
60
80
100
0
500
1000
1500
2000
2500
3000
3500
4000

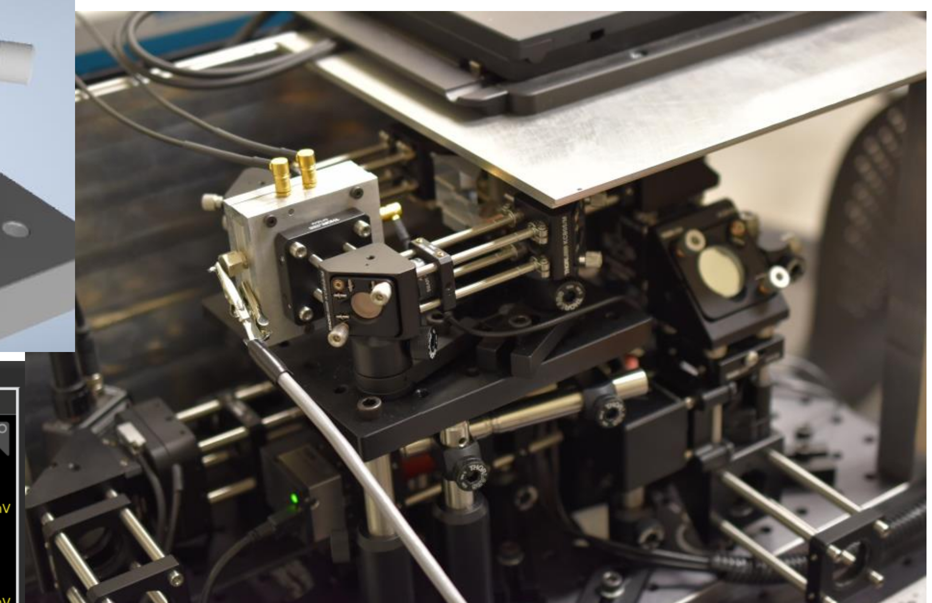

FFT, Grating = 60
0
20
40
60
80
100
0
500
1000
1500
2000
2500
3000
3500
4000


FFT, Grating = 120
0
20
40
60
80
100
0
500
1000
1500
2000
2500
3000
3500
4000

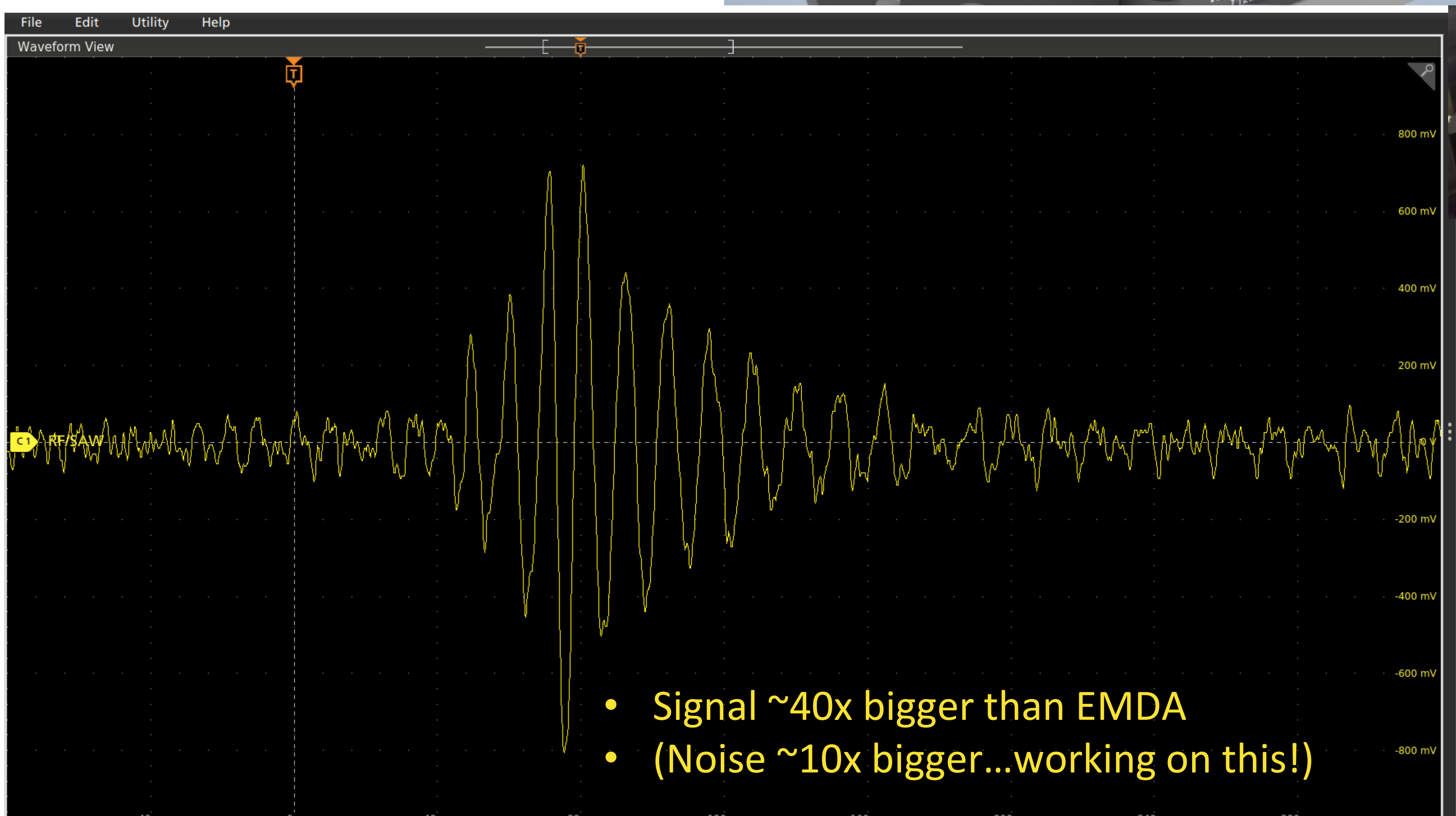
Detector (Gen 2 - new design)







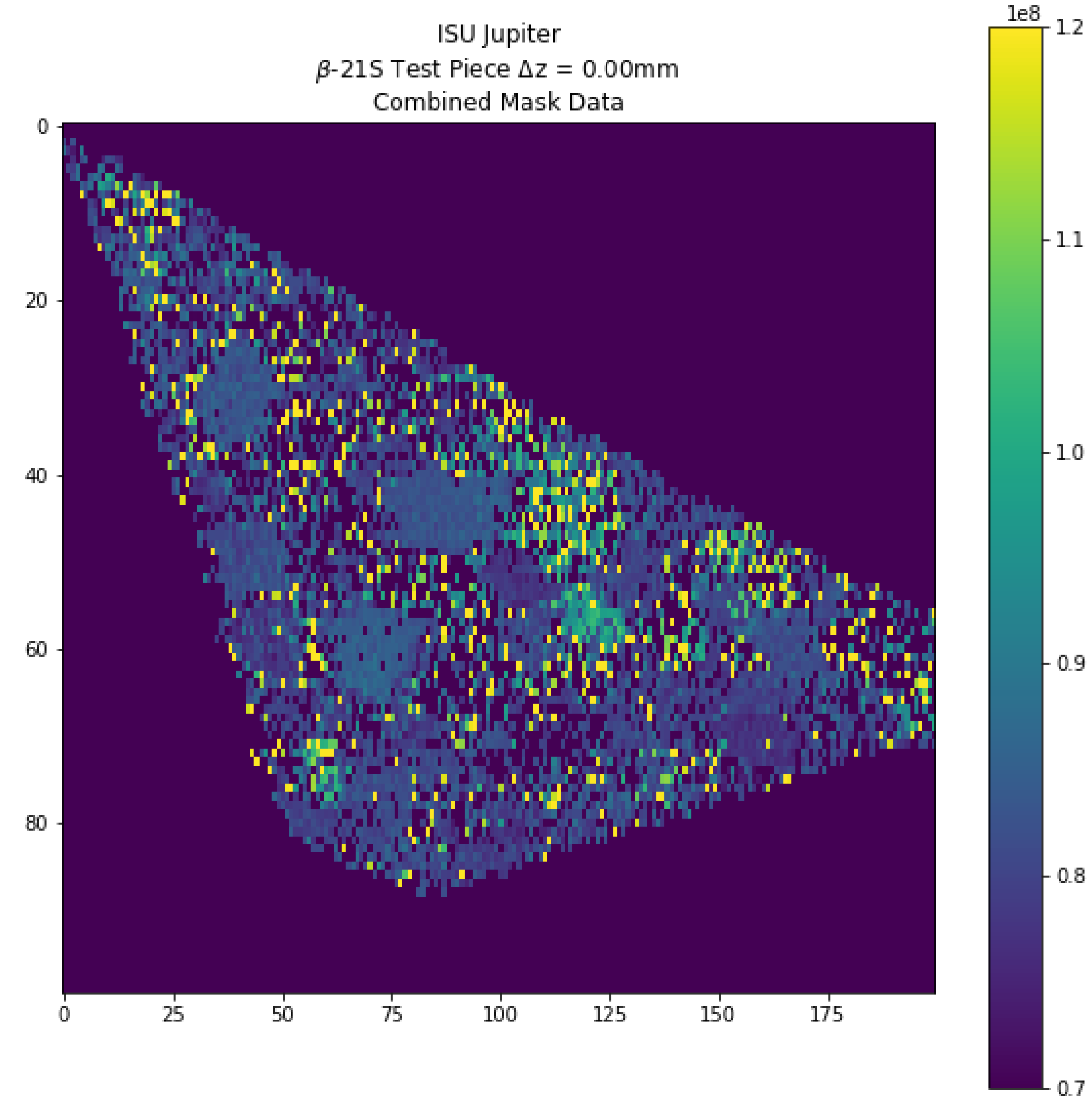
November



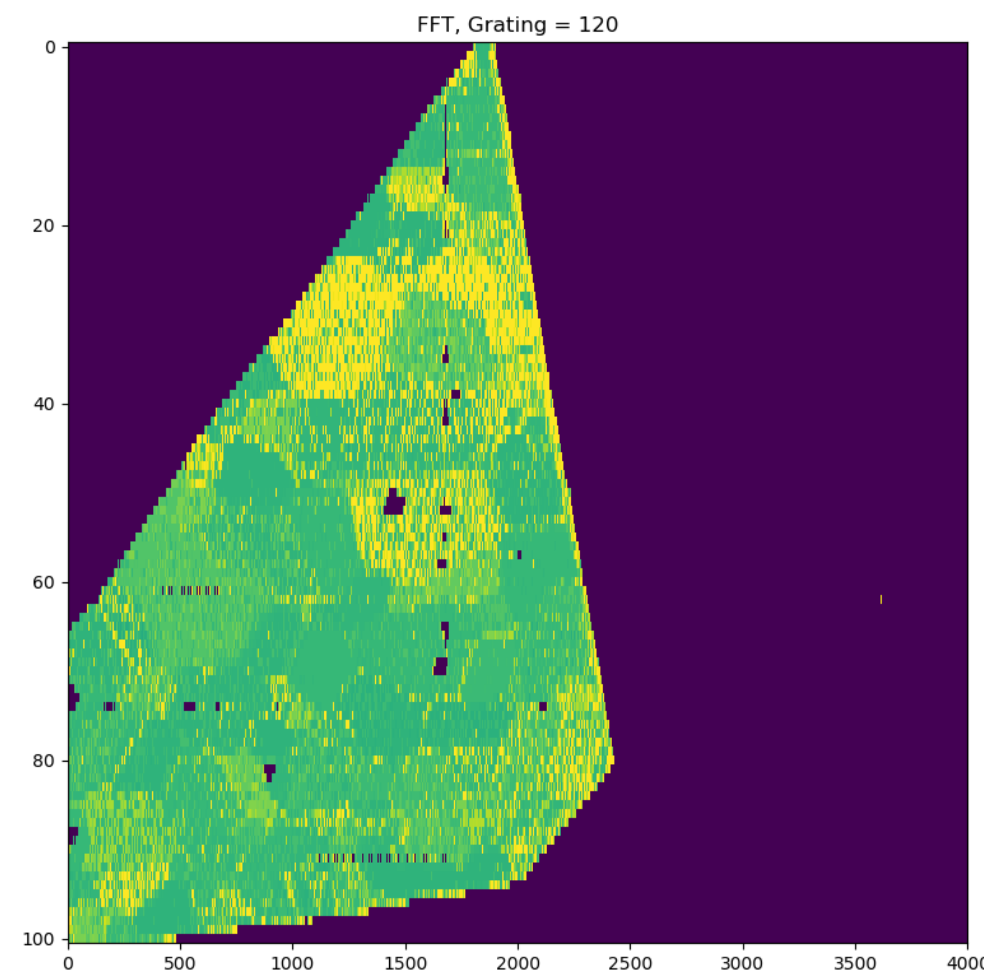
- Signal ~40x bigger than EMDA
- (Noise ~10x bigger…working on this!)

Present State-of-the-Art

- ISU Alpha system running, efforts being spent on improving resolution, data acquisition and data transfer rates
- Current RAW data is ~15 MB/mm^2
- Potential to move away from oscilloscope storage for data



1st scan acquired

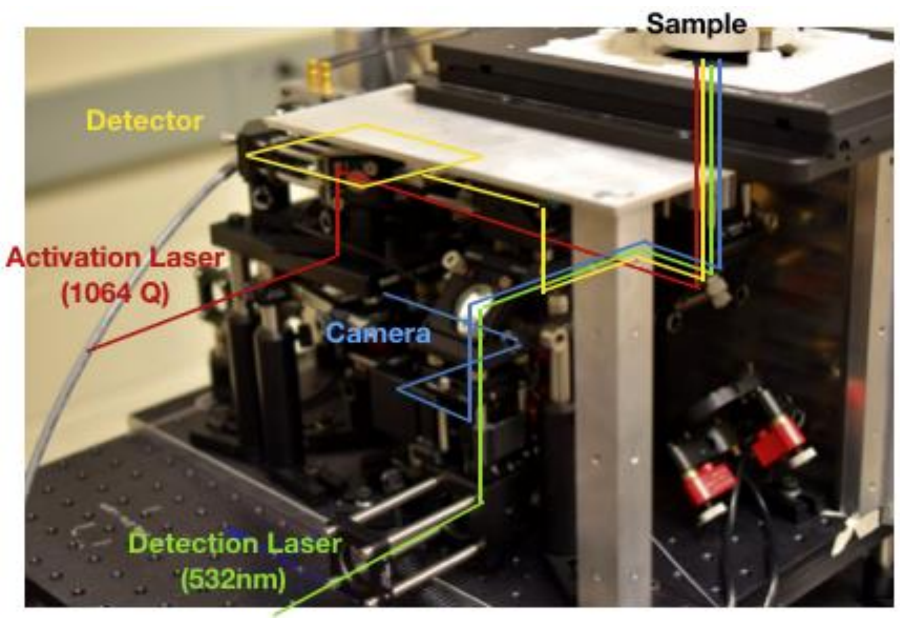


latest scan

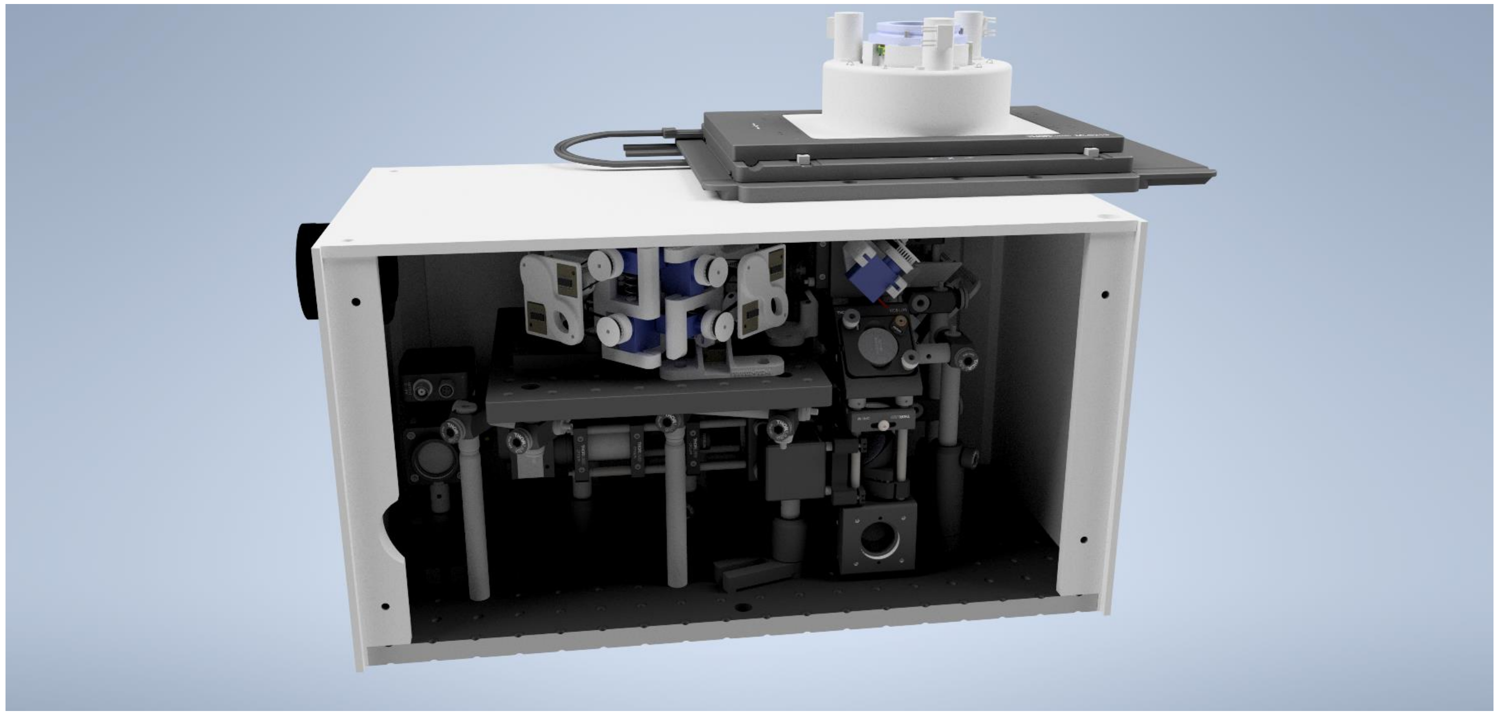
What is left…in the short term

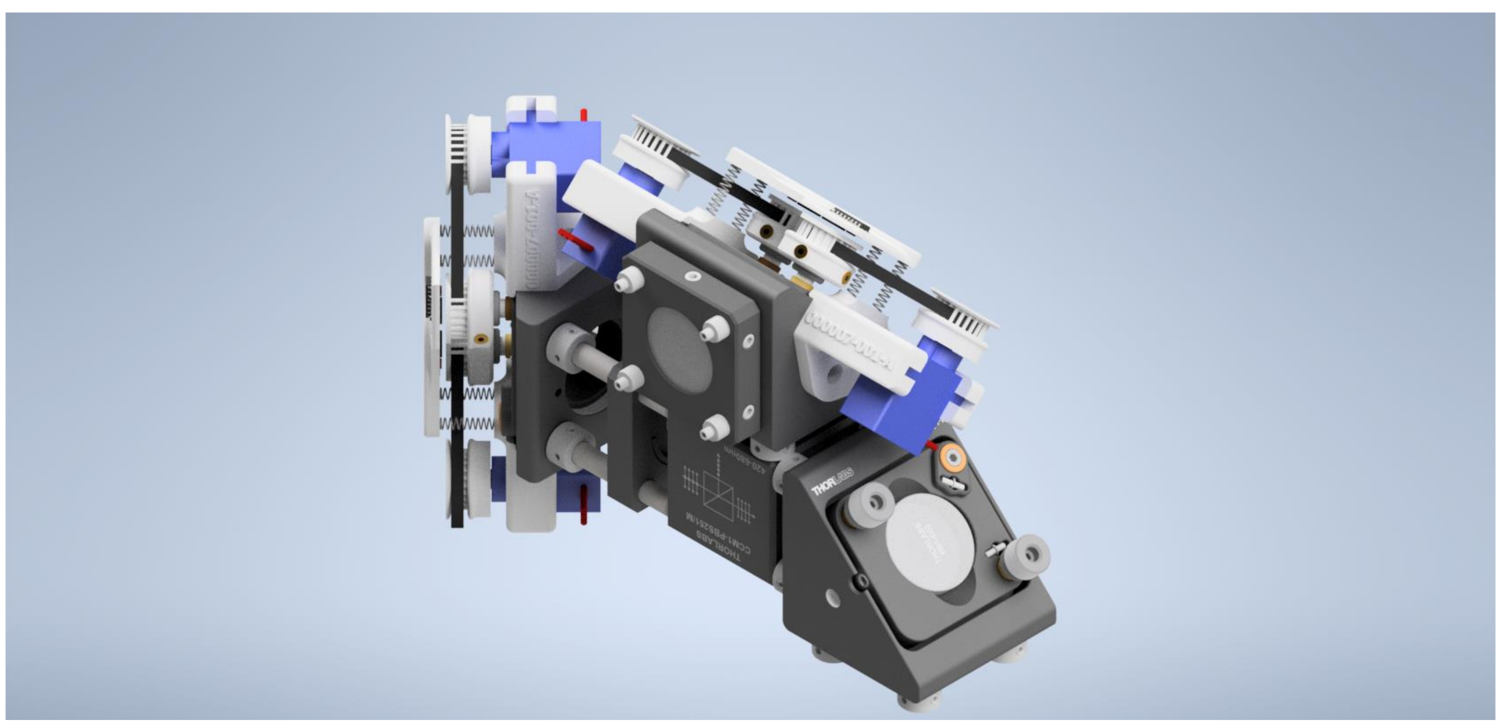
Left to resolve:

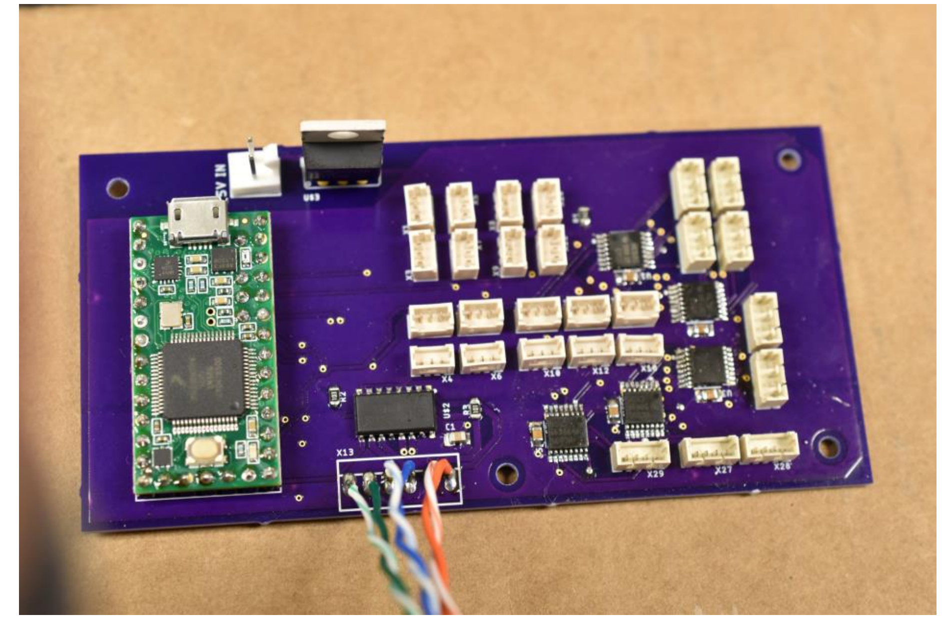
1. Taking the “Thomas” out of the system
    - Requires motorizing mirrors (alignments for every rotation … at the moment) - very difficult to do in an already very tight shoe box
    - Requires digital cameras for alignment
2. Make the system “Class 1” laser safe
3. Magnetic holders (done, as of yesterday)
4. Transition to fiber coupled lasers
5. (intermediate term) - moving from an oscilloscope to direct collection



Remotely Operable

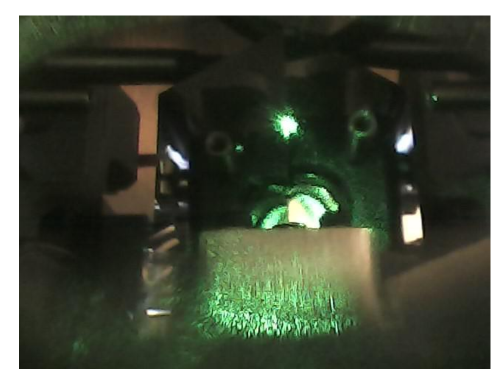


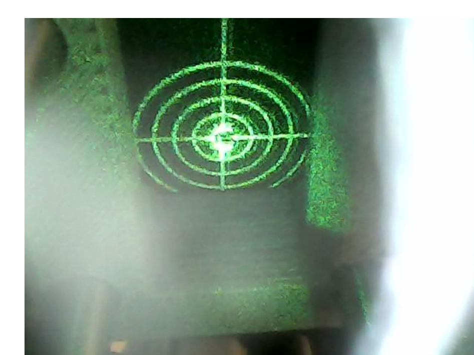




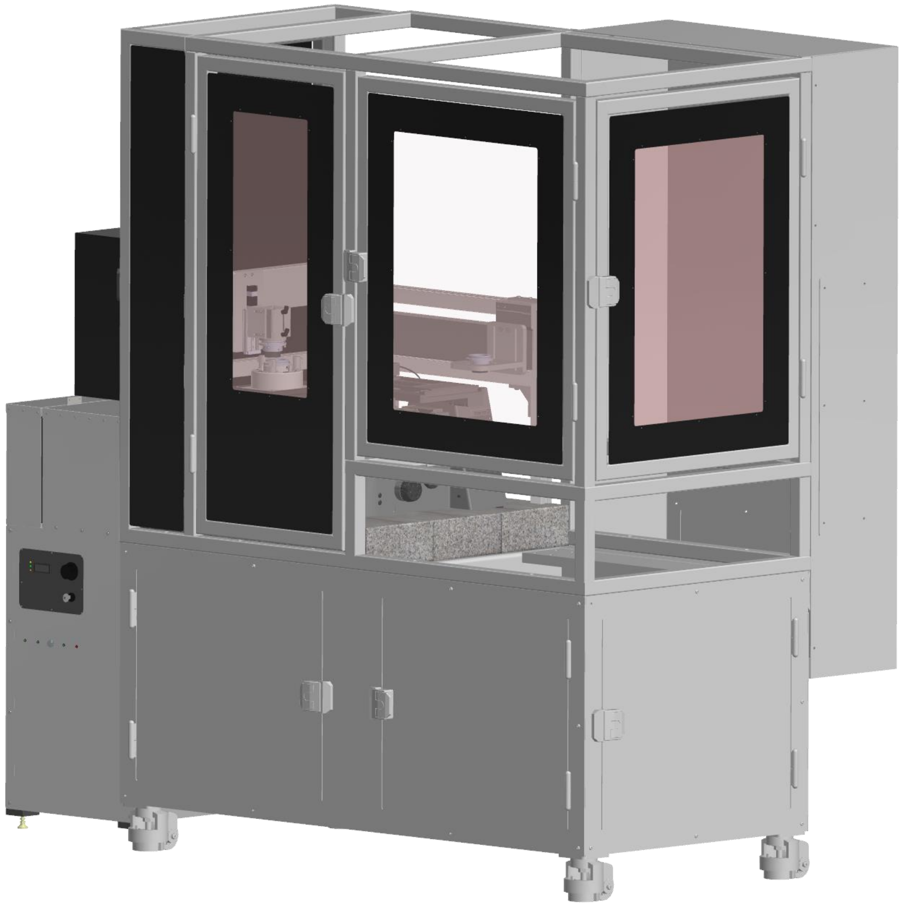
New motors, drivers, and sensors.

Can't be piezo-based.
- **Too noisy (electrically)**
- **Too big**





“Class 1”



What is left…in the long term

Future possibilities

1. Rough surfaces - possibly even in the "as built" condition
2. Non planar (i.e., curved) surfaces
3. Non linear analysis paths (i.e., for MSA of electronic devices)
4. Improved resolution (a "quantum leap" to 1um resolution?)
5. Time resolved experiments
6. Real-time determination (requires both a Gen-3 detector, laser upgrades, and clever databased approaches)

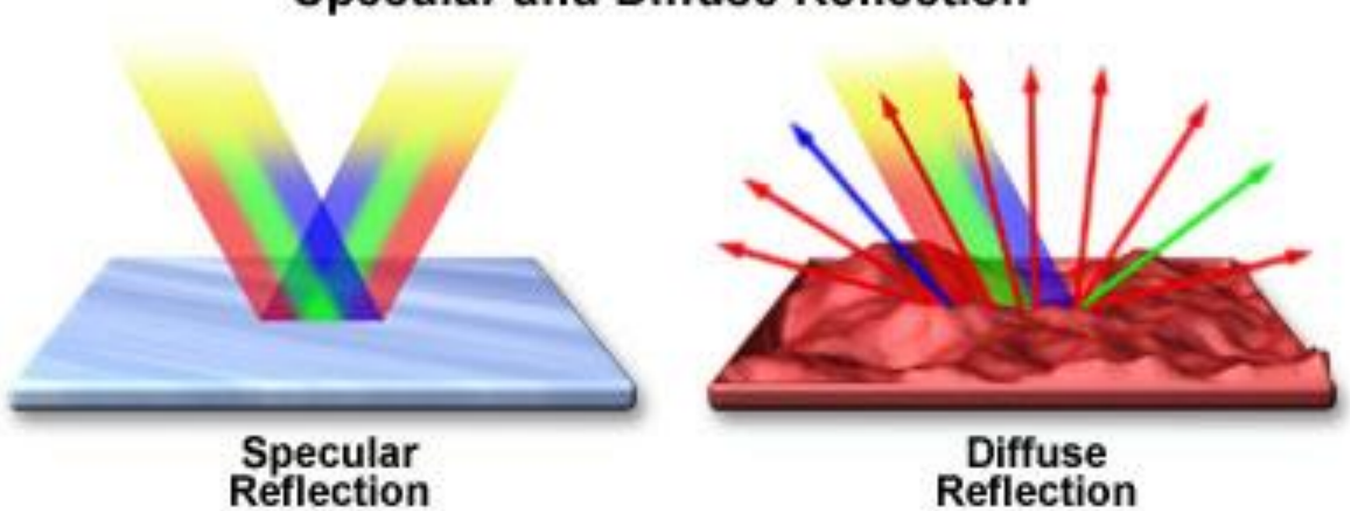
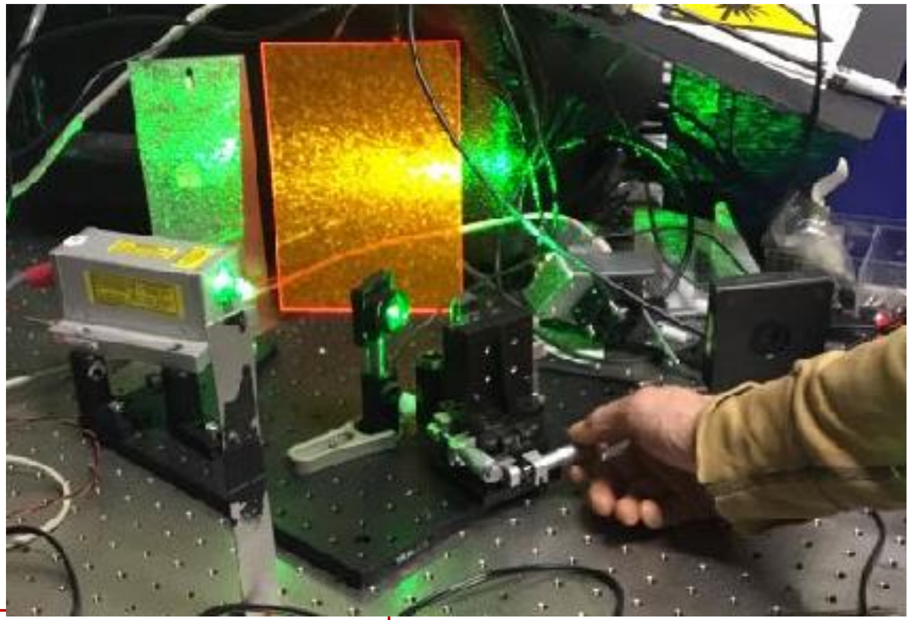
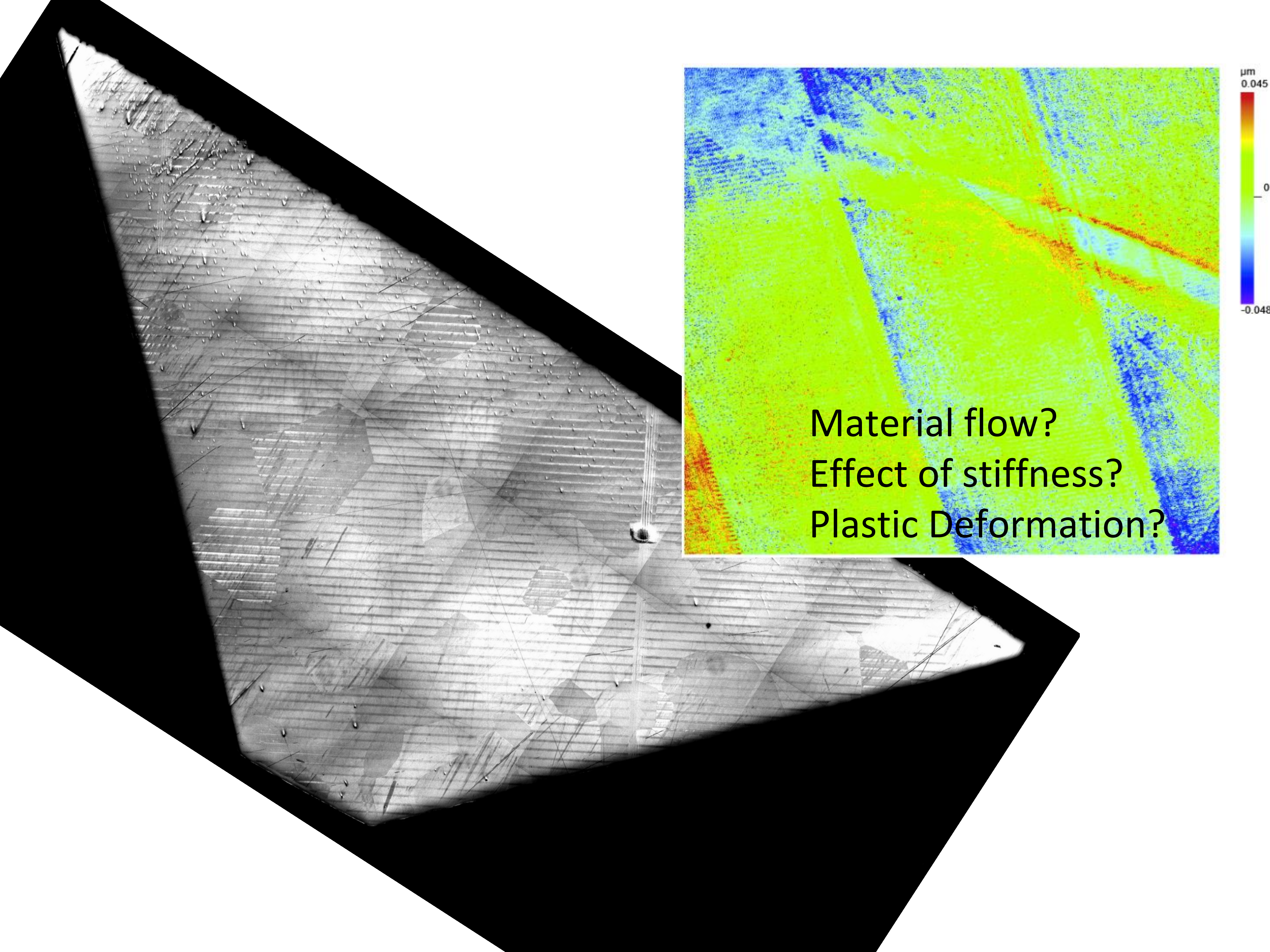


Figure 1




µm
0.045
0
-0.048
Material flow?
Effect of stiffness?
Plastic Deformation?

Bottom Line: Possibilities and Limitations

Possibilities:

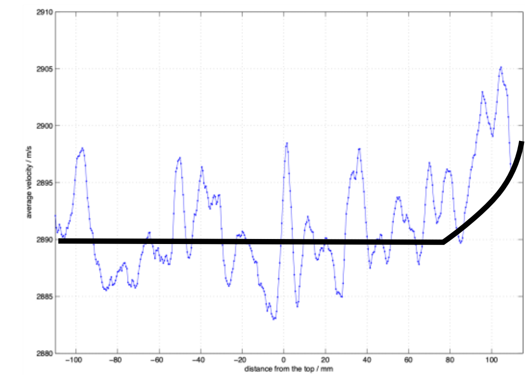
- Rapid orientation microscopy at large length scales (dm^2) and in 3D (cm^2)
- Time resolved experiments of dynamics
- Measure/map any _single_ variable that affects C_{ij} (including composition)

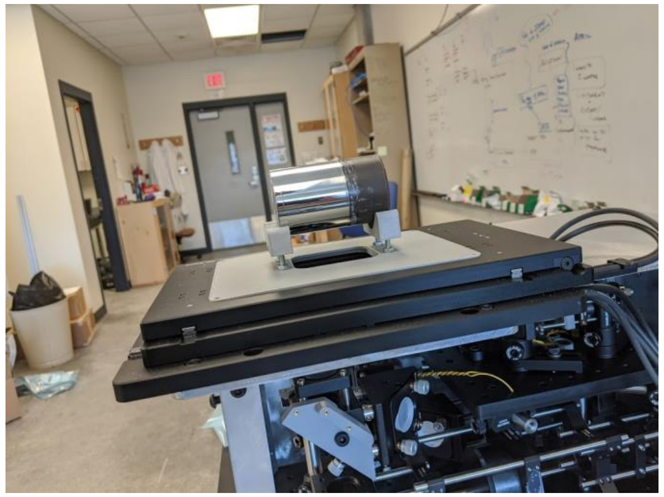
Probabilities:

- Orientation microscopy on rough surfaces (demonstrated in UK)
- Orientation microscopy on curved surfaces (theoretically possible)

Limitations:

- No split photo diode with sufficient bandwidth (resolved for now)
- Resolution (but a higher resolution should be possible)
- Data and bandwidth is a challenge (but solvable)
- Manufacturing infrastructure (resolving…but it takes time)
- Sparsity in scientists
- Multiple variables will convolve the signal





Thank you!!

pcollins@iastate.edu