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A New Paradigm in Metal Surface Preparation for High-Throughput Materials Characterization

Elliott Fowler
Sandia National Laboratories

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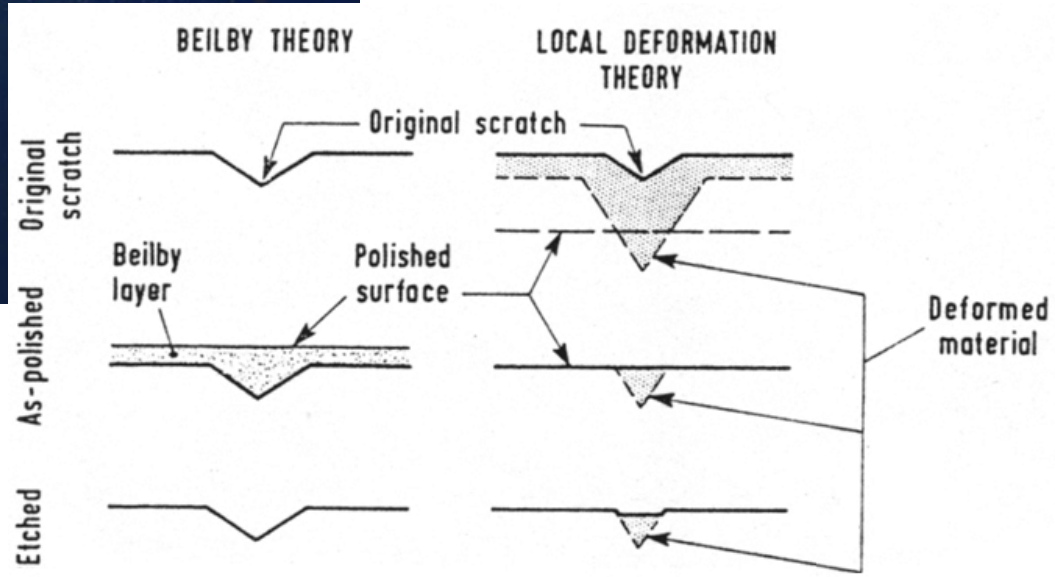
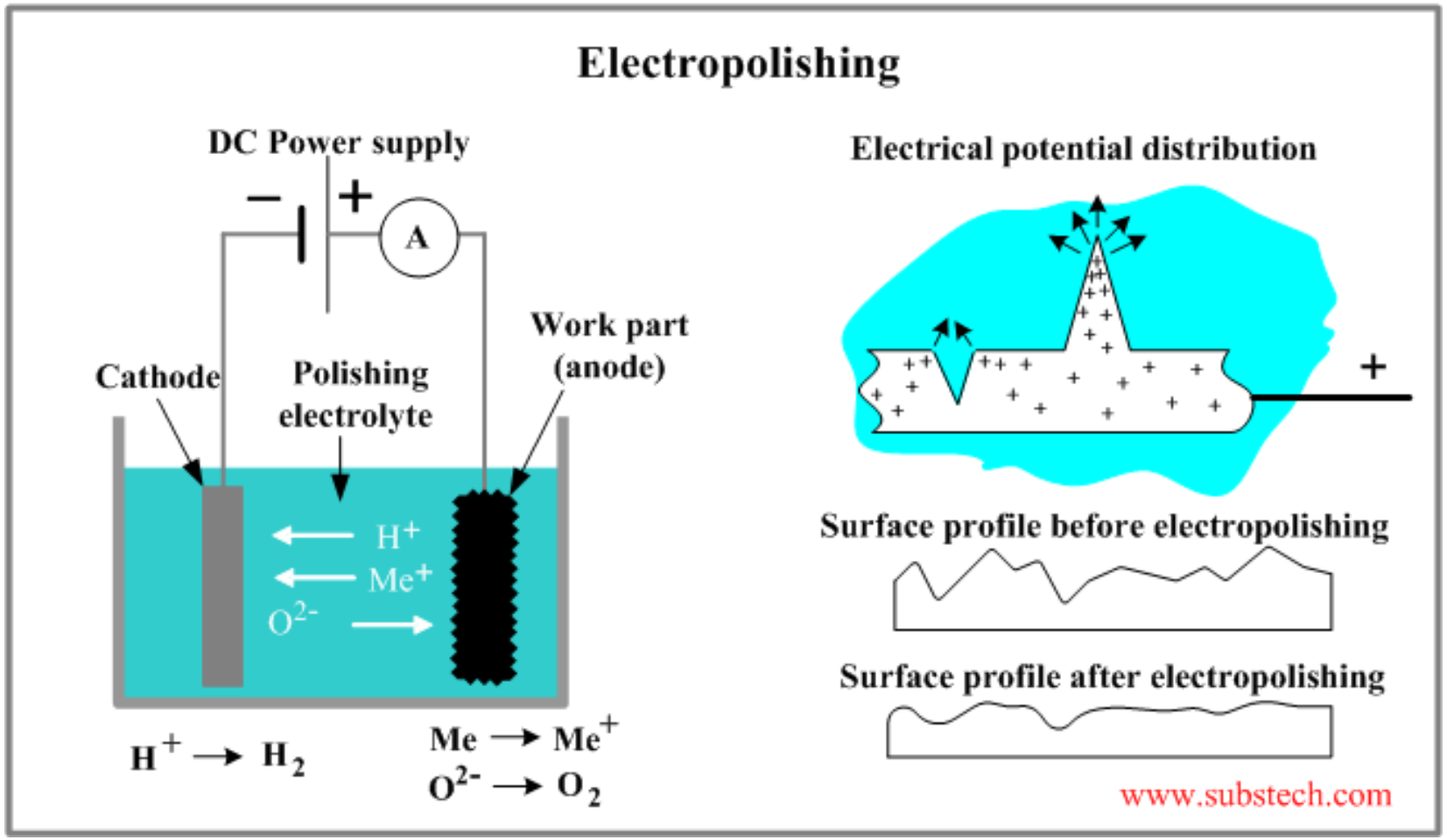
Manual Metallographic Polishing is Slow, Artisanal and Can Leave a Surface Deformation Layer



Alternatives to Manual Polishing

"Wet" Electropolishing

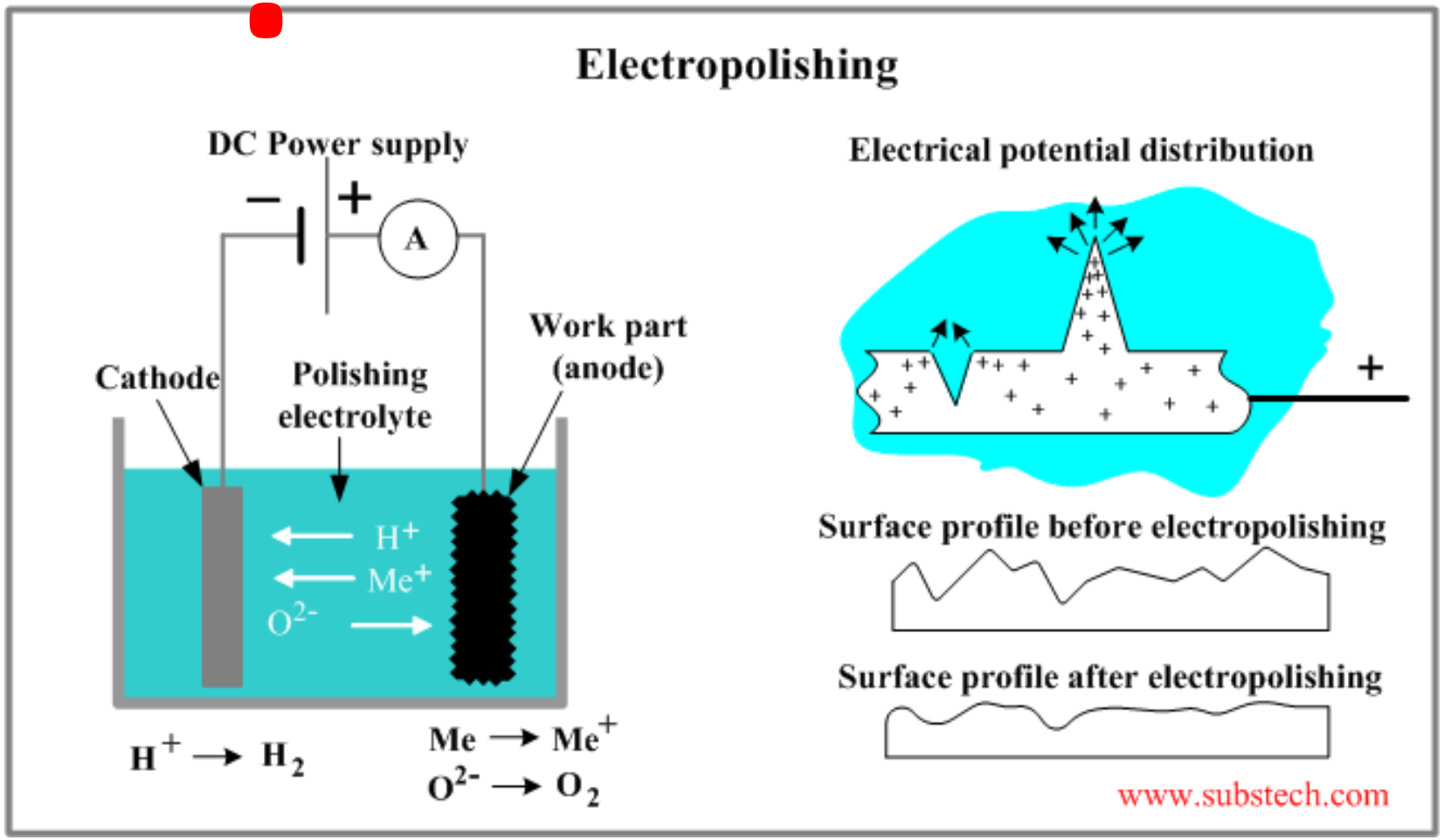


- Liquid contacts entire surface
- All surfaces oxidized
- Low discrimination

Alternatives to Manual Polishing

?

Conventional Electropolishing



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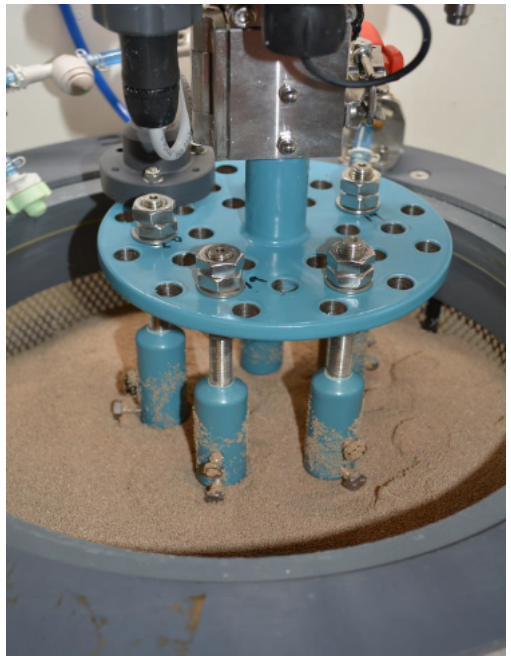
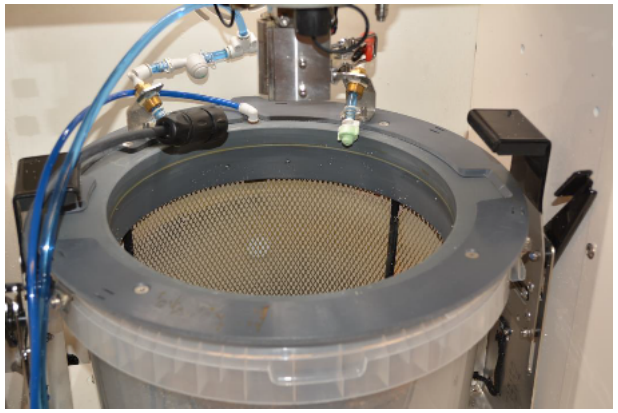
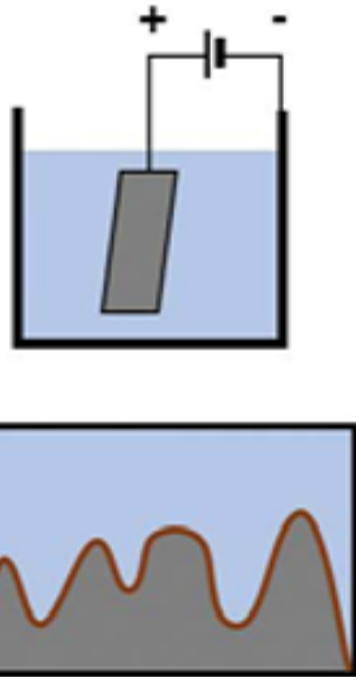
Alternatives to Manual Polishing





Alternatives to Manual Polishing

Conventional Electropolishing



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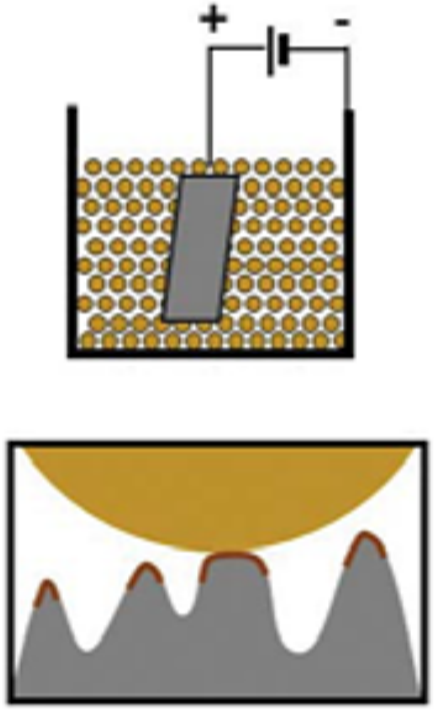
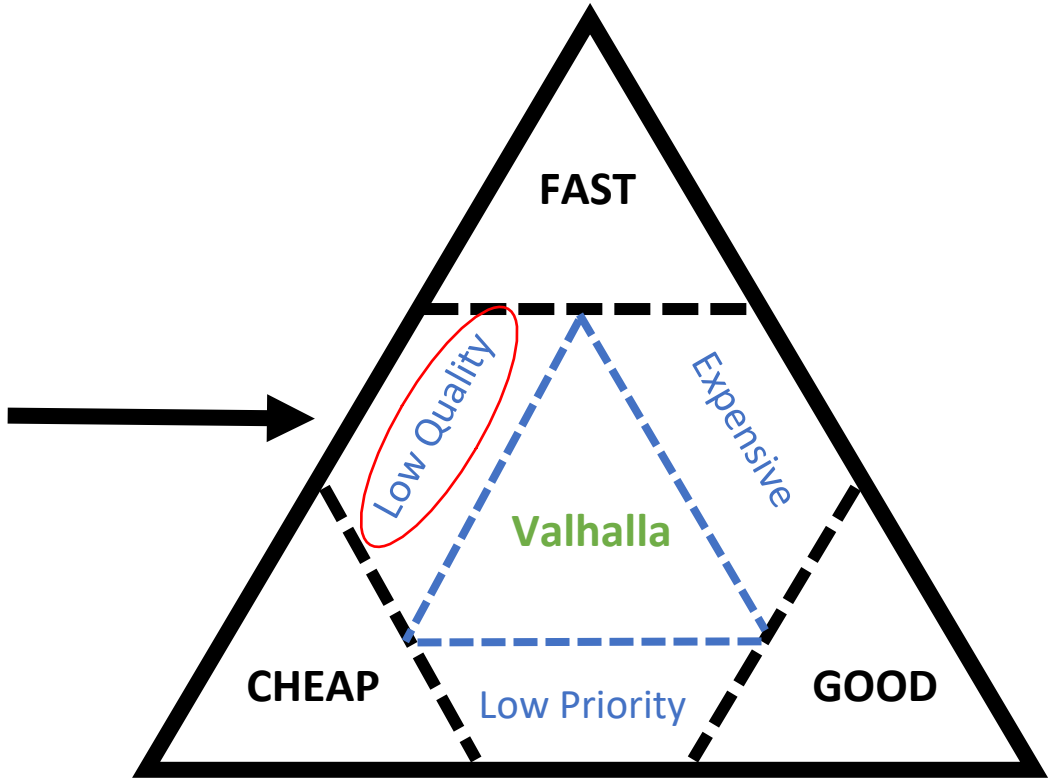
Dry Electropolishing



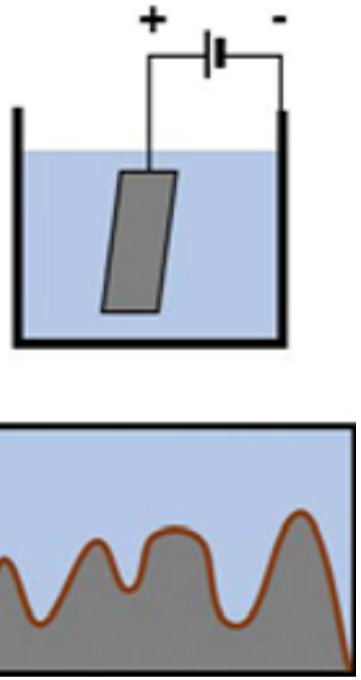
- Contact surface peaks
- Localized oxidation
- Selective removal

The Iron Triangle



Alternatives to Manual Polishing

Conventional Electropolishing



- Liquid contacts entire surface
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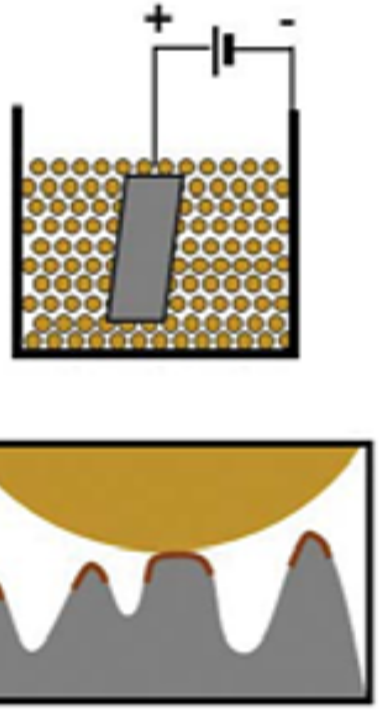
- Low discrimination

Dry Electropolishing



- Contact surface peaks
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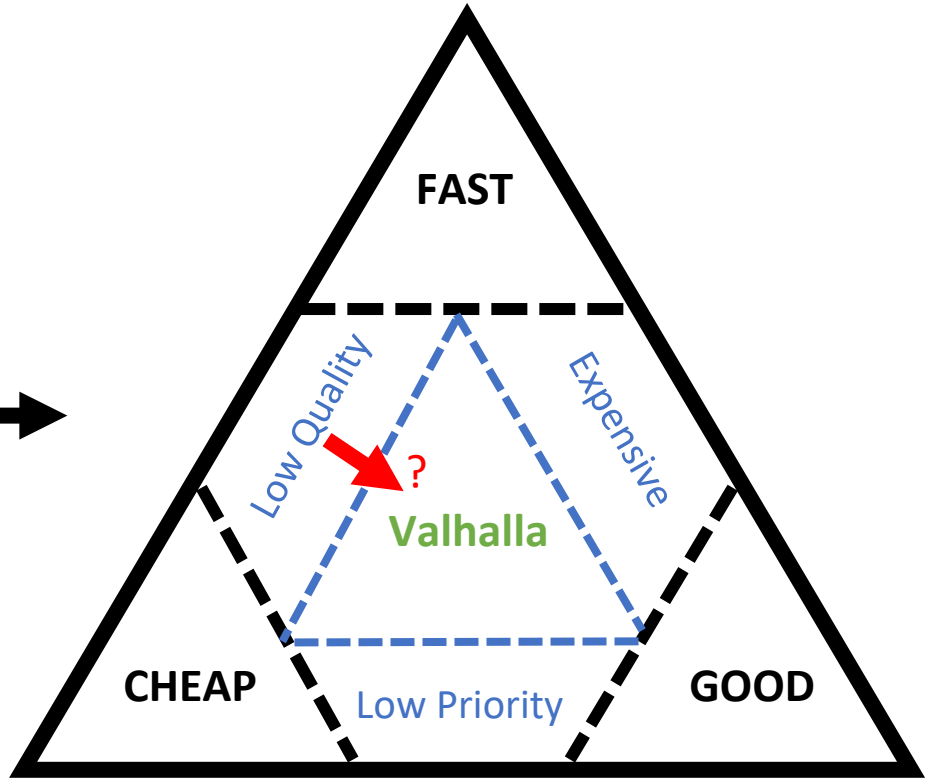
The Iron Triangle



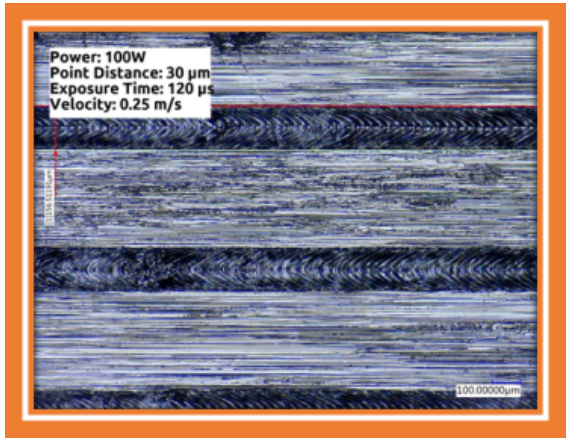
Can We Reach the Valhalla of Metallographic Surface Preparation?

Melt Pool Imaging of LPBF Laser Scans on Kovar

Team –

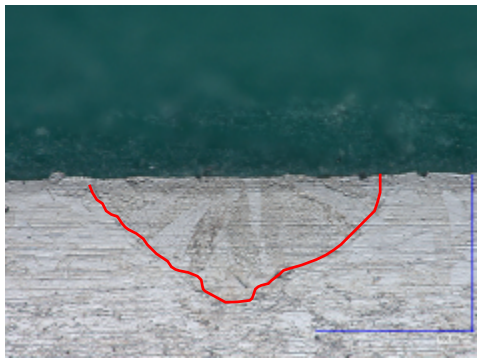
Elliott Fowler (1819), Kyle Johnson (1558), Dale Cillessen (7585), Jay Carroll (1851), Tim Ruggles (1819), Luis Jauregui (1819)

TECHNICAL CONCEPT

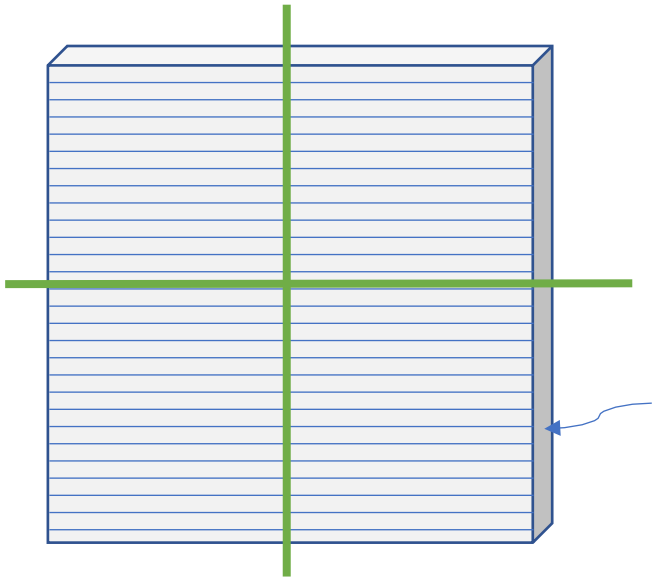


480 Lines
240 Unique Process Conditions

A B C D



Melted Metal



Metlab considerations:

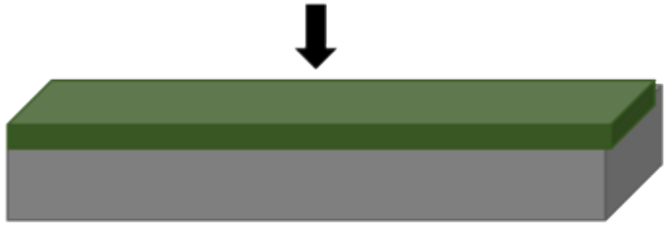
- 240 line scans across plate surface
- Analyzing both edges requires 480 welds to be prepared
- Plate dimensions are too large for standard prep in Metlab so requires cutting
- Plate dimensions necessitate epoxy mounting as there is no way to fixture the plate during polishing

Standard prep step	Time estimate for four pucks
Cut (green lines) to fit inside four 1.5-2” pucks	1 hour
Mount	12 hour cure time
Grind	0.5 hour
Diamond polish	2 hours
Vibratory polish	8 hours
Etch	1 hour
Image and measure weld dimensions	7 minutes/weld*480 = 56 hours

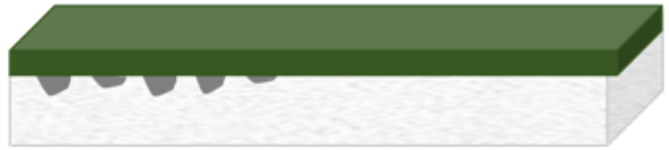
Total time for 480 welds per standard prep procedure = **80.5 hours**

This estimate applies to 1 plate only (4 mounts). Polishing is done in sets of 6 mounts. Two plates would produce 8 mounts, so this estimate would need to be doubled.

As-Cut Kovar Piece (w/ Line Scans)



10 minute Ar plasma etch, mount in epoxy



30 minute grind, 10 minute dry electropolish, 10 sec. etch

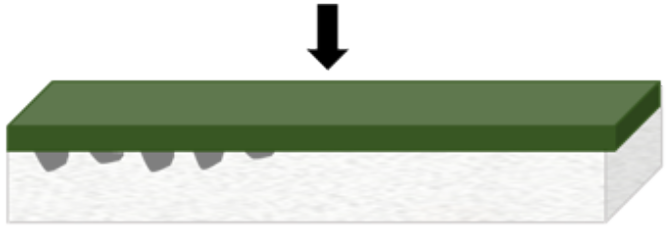


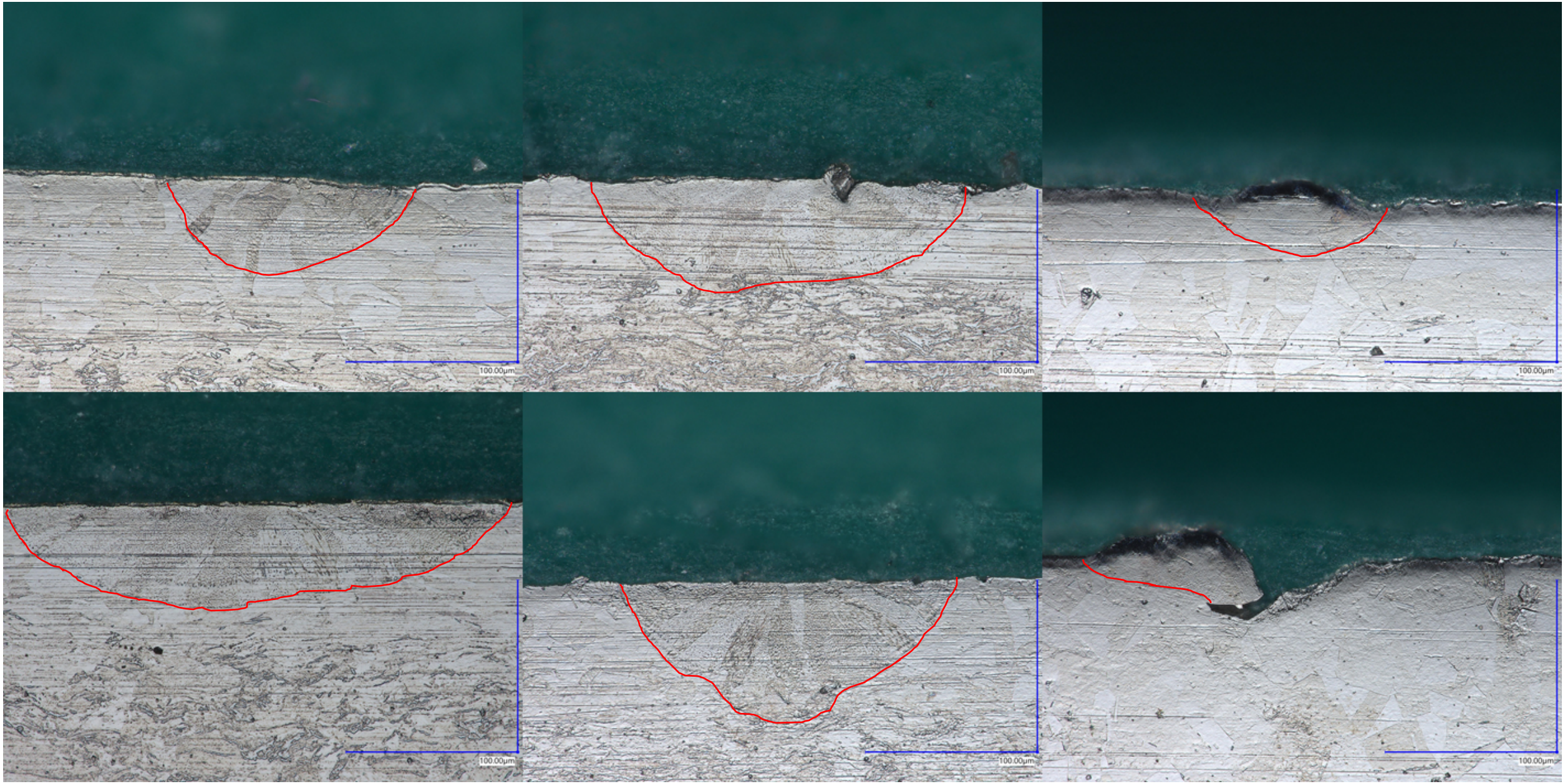
Image at 1000x

Method → Task ↓	Traditional Preparation	HT MP Imaging Prep
Cut	1 hrs	0.5 hrs
Mount	12 hrs	0.08 hrs
Grind	0.5 hrs	0.5 hrs
Polish	10 hrs	0.2 hrs
Etch	1 hr	0.003 hrs
Imaging	56 hrs	16 hrs
Total	80.5 hours	~17.5 hours
Total Improvement	-	**4.6x**

Basis – Per set of 4 samples, representing 1 cross-section of 480 line scans

Reduced Prep Time from 24.5 hours to ~1.5 hours and can scale up for ‘free’

~16x improvement in prep time


100.00µm
100.00µm
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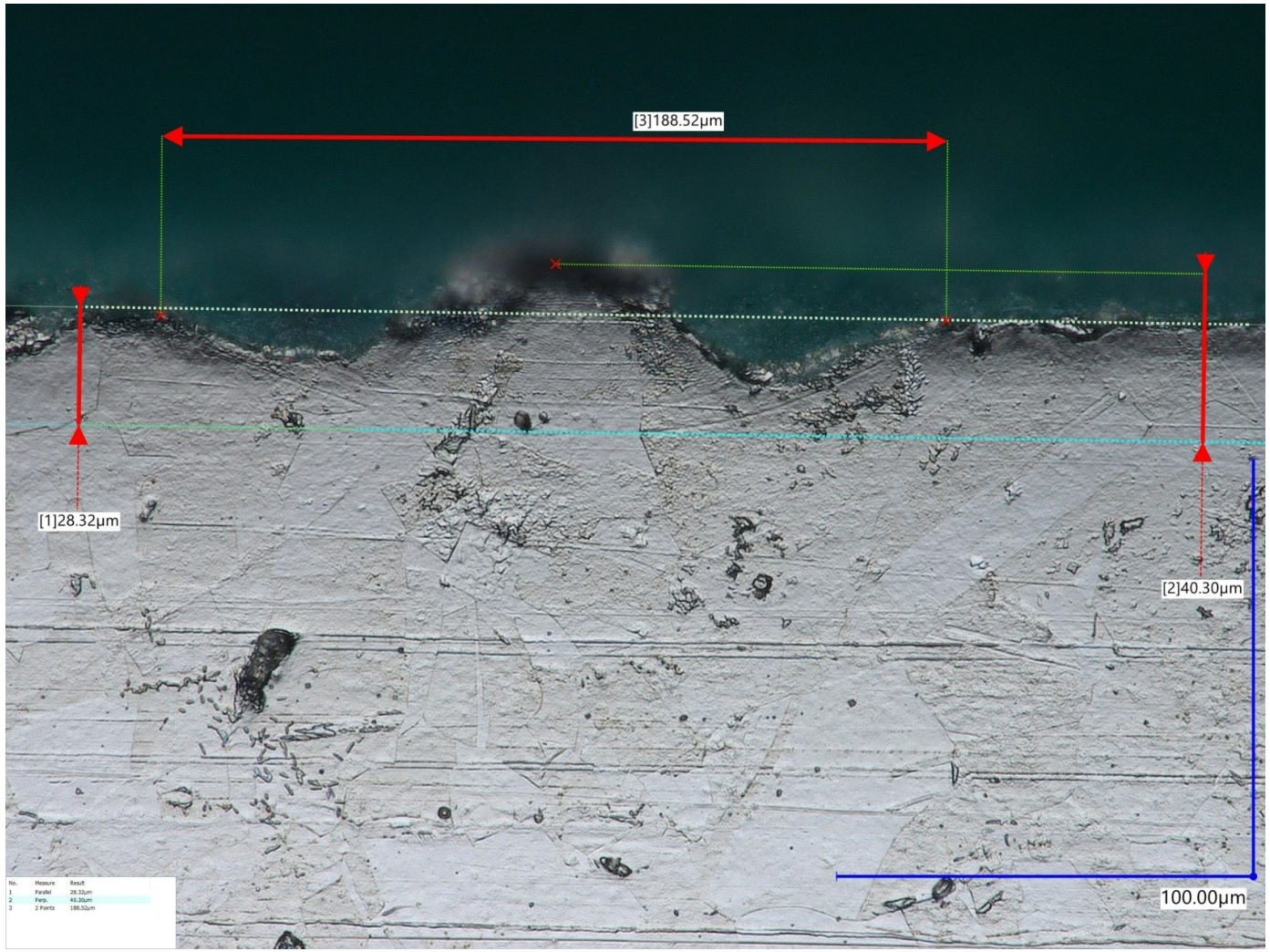
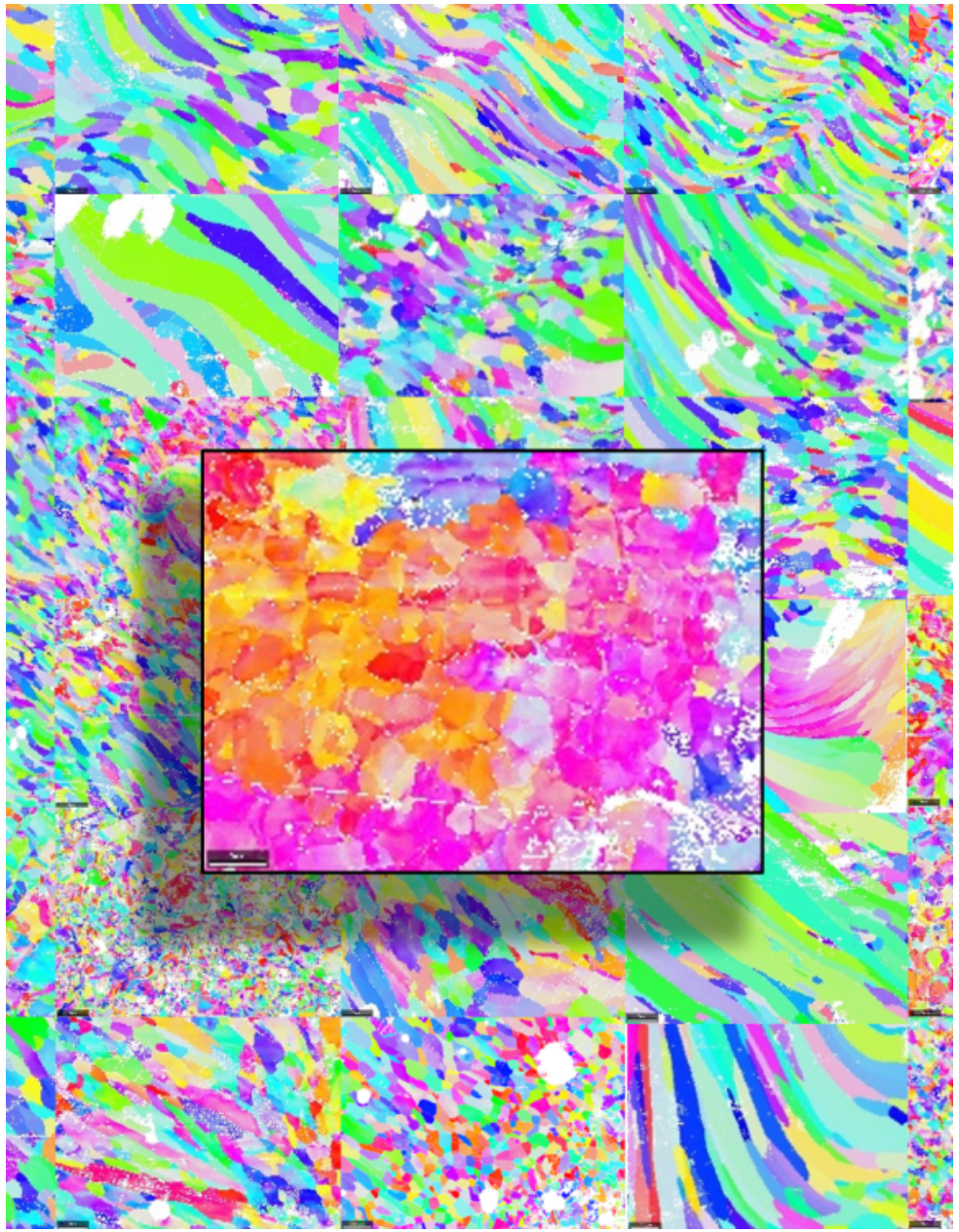
100.00µm



Labeler	Measurement		
	Depth	Peak to Trough	Width
1	28.32	40.3	188.52
2	27.78	45.15	180.78
3	26.34	44.87	189.05

HIGH THROUGHPUT EBSD OF AM KOVAR

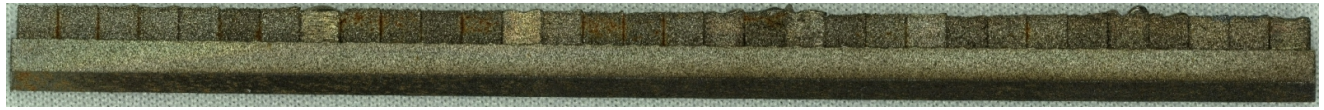


<u>Team –</u>

Elliott Fowler (1819), Kyle Johnson (1558), Dale Cillessen (7585), Jay Carroll (1851), Tim Ruggles (1819), Luis Jauregui (1819)

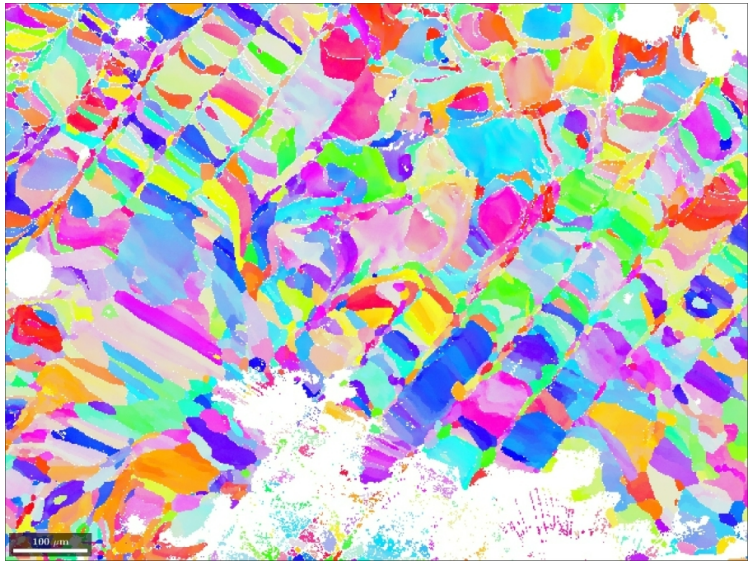
TECHNICAL CONCEPT

As-Printed, LPBF/AM Printed Kovar Sample Bar (EDM Cut from Platen)
32, 3 mm x 3 mm samples per bar; 8 bars per platen; ~250 samples per print



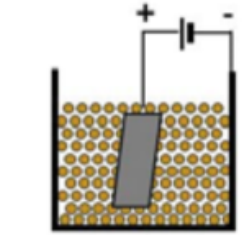
GOAL

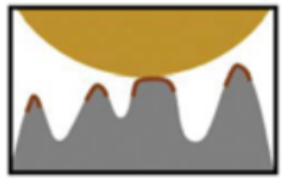
As-Printed Surface to EBSD Patterns in ~24 Hours



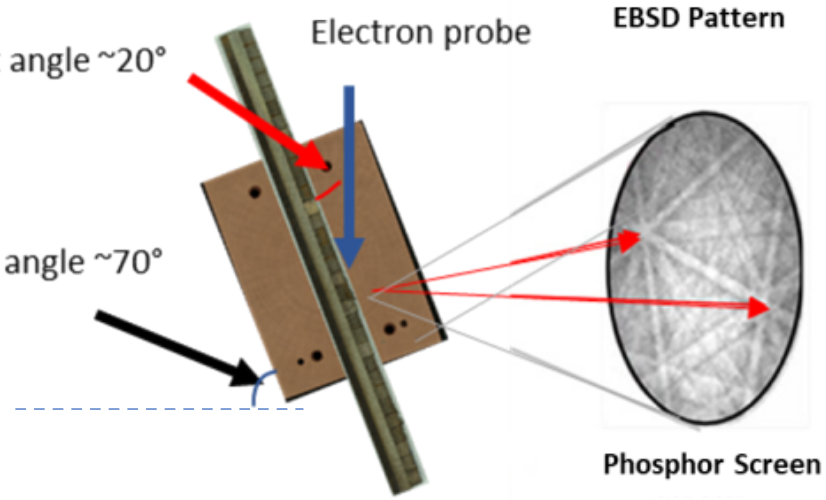
Lots of Math

DLyte Electropolishing





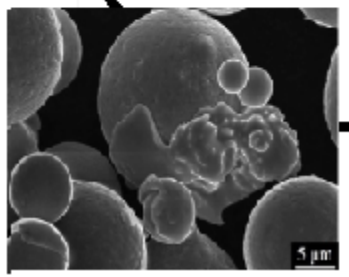
- Contact surface peaks
- Localized oxidation
- Selective removal

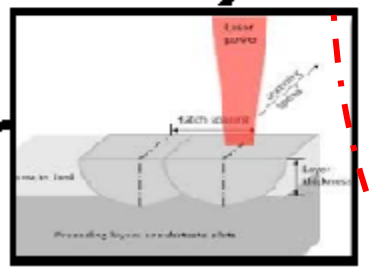


EBSD MAPPING IS ONE OF THE MOST COMPREHENSIVE MICROSTRUCTURE DESCRIPTORS AVAILABLE IN THE MATERIALS CHARACTERIZATION TOOLBOX

PROCESS

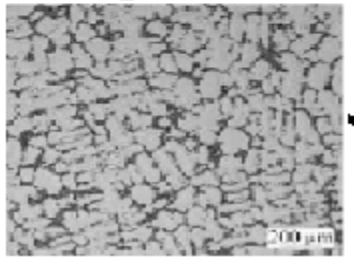
* Powder Production
* Additive Manufacturing
* Post HIP, Heat Treatment, etc.

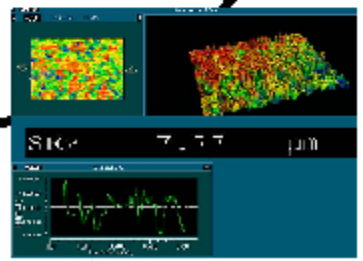




STRUCTURE

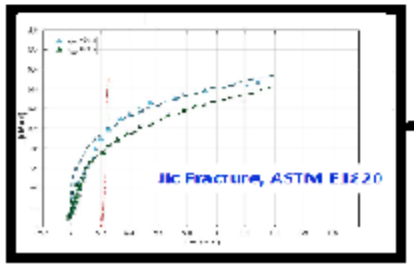
* Microstructure & Texture
* Dislocations, twins, crystal defects
* Macrostructure
* Macrodefects - Porosity, unmelted particles, lack of fusion seams
* Form, finish & waviness

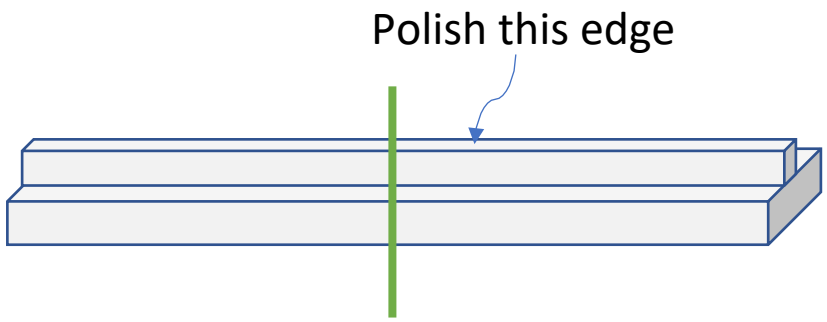




PROPERTIES

* Mechanical properties
- UTS, US, %Elong, etc.
- Creep & Fatigue
- Residual stress
- Toughness





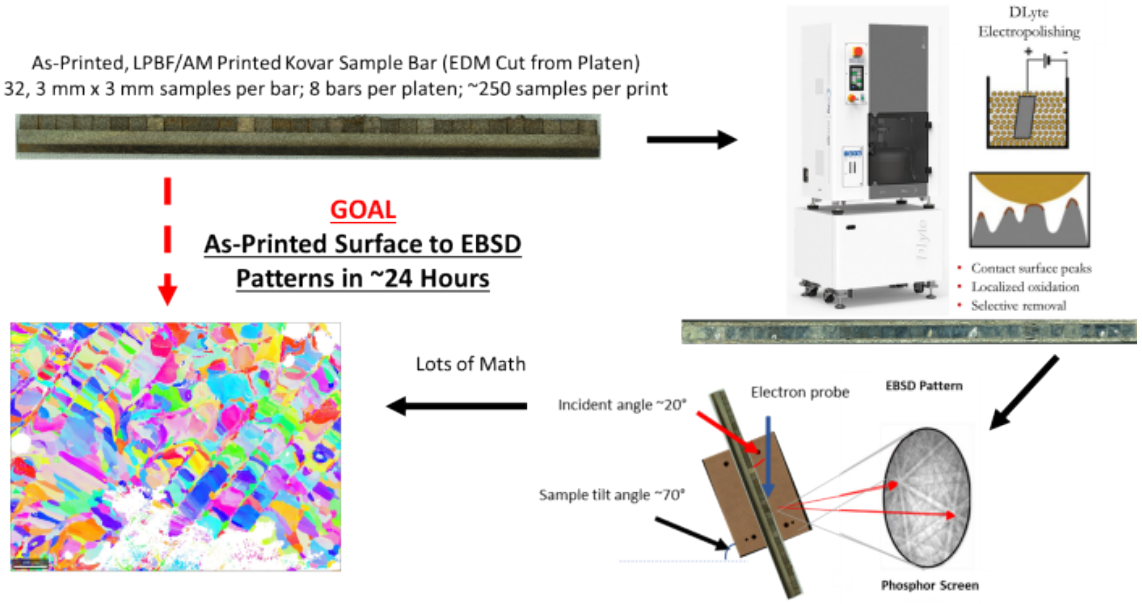
Metlab considerations:

- Plate dimension is too large for standard prep in Metlab so requires cutting
- Bar shape necessitates epoxy mounting as there is no way to fixture the part during polishing
- Cutting is undesirable due to material loss from blade kerf thickness. Entire continuous length cannot be analyzed simultaneously if cut and mounted.

Standard prep step	Time estimate for two pucks
Cut (green line) to fit inside two 1.5-2” pucks	1 hour
Mount	12 hour cure time
Grind	0.5 hour
Diamond polish	2 hours
Vibratory polish	8 hours
	Total time for 1 bar = **23.5 hours**

EBSD Mapping (one by one) – 1 hour setup, 0.5 hrs per map, 16 hours per bar – if mitigated with overnight mapping would take 2x sessions to complete (2 pucks per bar)

This estimate applies to a sample set ranging from 1- 3 bars (2 mounts - 6 mounts). Polishing is done in sets of 6 mounts. The differential of prep time required for 2 mounts versus 6 mounts is negligible.



Method → Task ↓	Traditional Preparation	HT EBSD Prep
Cut	12 hrs	-
Mount	12 hrs	-
Grind	12 hrs	1 hrs
Polish	40 hrs	1.5 hrs
EBSD (overnight runs)	24 hrs	6 hrs
Total	100 hours	8 hours
Total Improvement	**-**	**12.5x**

Basis – 1 "Set" = 3 Bars of 32 Samples and 1 Bar of 28 Samples or 124 Samples

Additional Note – Since Samples in HT Method are Never Cut or Mounted, Additional HT analysis (e.g. - Mech. Properties) Can be Easily Completed

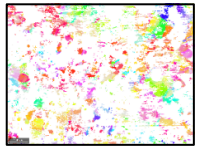
Reducing a process that takes 2 weeks for preparation (pre-EBSD) to one that takes 2.5 working hours.

"Prep-Only" Improvement - ~32x

AGS = Average Grain Size ; AAR = Average Aspect Ratio

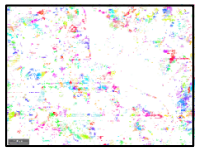
**All maps x-ipfs, FCC

1



2

AGS – 5.30 µm
AAR – 1.54

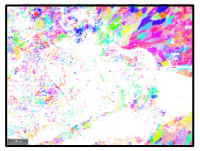


AGS – 3.83 µm
AAR – 1.50

3

AGS – 4.44 µm
AAR – 1.58

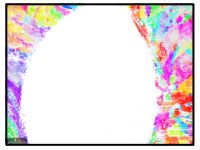
4



AGS – 4.75 µm
AAR – 1.35

5

AGS – 7.50 µm
AAR – 1.47



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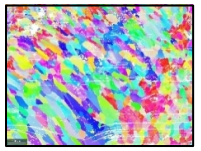
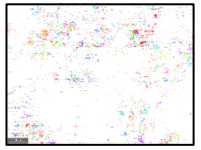


AGS – 11.67 µm
AAR – 1.93

7

AGS – 3.17 µm
AAR – 1.36



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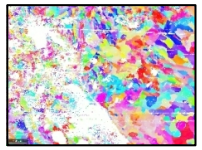
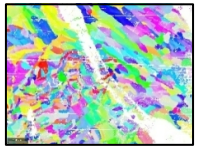


AGS – 6.53 µm
AAR – 1.51

9

AGS – 9.01 µm
AAR – 1.86

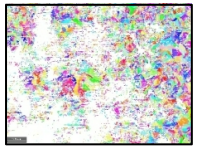


10

AGS – 7.18 µm
AAR – 1.94

11

AGS – 4.18 µm
AAR – 1.52

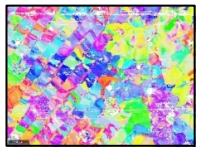


12

AGS – 7.07 µm
AAR – 1.73

13

AGS – 11.22 µm
AAR – 1.70



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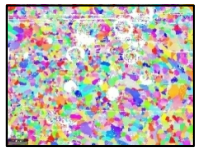
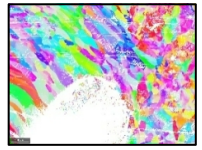


AGS – 7.38 µm
AAR – 1.61

15

AGS – 11.16 µm
AAR – 1.40



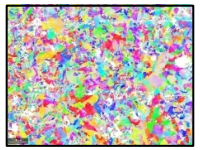
16

AGS – 5.23 µm
AAR – 1.78

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16

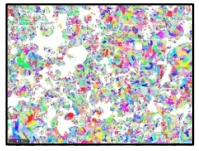
17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32

17



AGS – 5.87 µm
AAR – 1.65

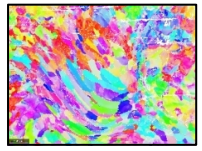
18



AGS – 4.29 µm
AAR – 1.58

19

AGS – 12.35 µm
AAR – 1.99



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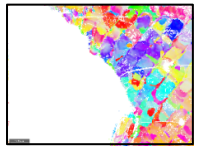
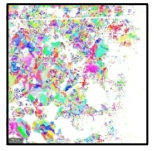


AGS – 10.25 µm
AAR – 1.57

21

AGS – 4.24 µm
AAR – 1.52



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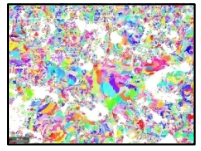
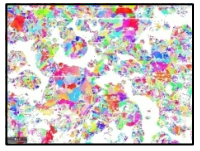


AGS – 4.69 µm
AAR – 1.59

23

AGS – 4.65 µm
AAR – 1.58



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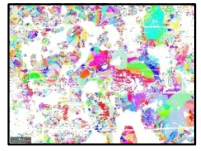
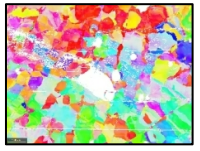


AGS – 4.69 µm
AAR – 1.49

25

AGS – 13.21 µm
AAR – 1.60



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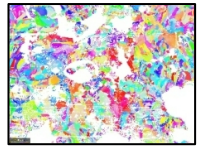
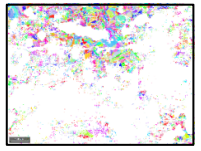


AGS – 5.94 µm
AAR – 1.64

27

AGS – 3.94 µm
AAR – 1.45



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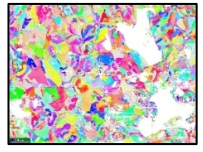
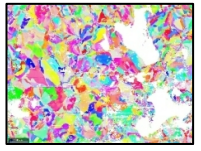


AGS – 6.30 µm
AAR – 1.81

29

AGS – 7.50 µm
AAR – 1.96



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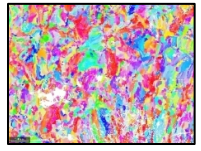
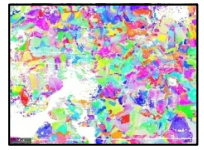


AGS – 7.27 µm
AAR – 1.86

31

AGS – 6.46 µm
AAR – 1.69



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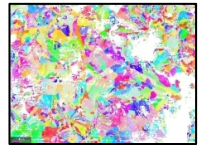
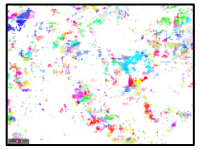


AGS – 6.21 µm
AAR – 1.69

AGS = Average Grain Size ; AAR = Average Aspect Ratio

**All maps x-ipfs, FCC

1



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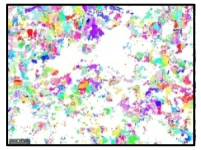
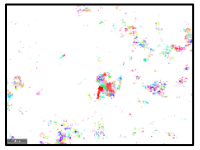


AGS – 4.59 µm
AAR – 1.48

3



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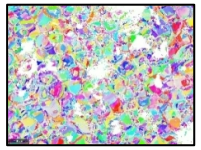
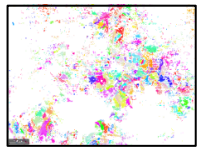


AGS – 4.99 µm
AAR – 1.66

5



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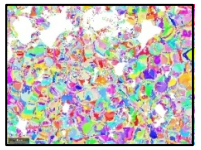
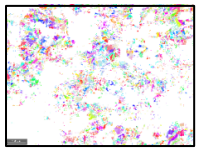


AGS – 3.76 µm
AAR – 1.46

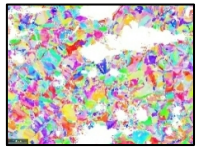
7



8

AGS – 4.99 µm
AAR – 1.82

9



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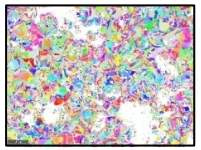
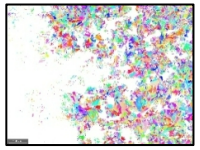


AGS – 4.41 µm
AAR – 1.38

11



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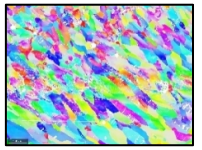
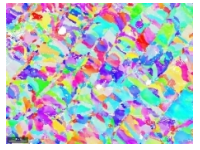


AGS – 3.95 µm
AAR – 1.48

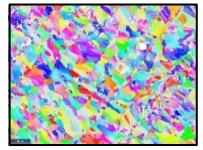
13



14

AGS – 6.24 µm
AAR – 1.81

15



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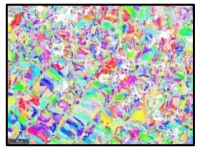


AGS – 4.22 µm
AAR – 1.60

AGS – 10.65 µm
AAR – 1.99

AGS – 11.22 µm
AAR – 1.96

AGS – 5.25 µm
AAR – 1.92

AGS – 5.22 µm
AAR – 1.73

AGS – 4.65 µm
AAR – 1.77

AGS – 13.51 µm
AAR – 1.83

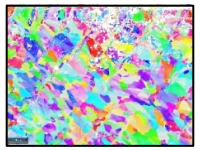
AGS – 5.57 µm
AAR – 1.84

AGS – 5.83 µm
AAR – 1.86

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16

17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32

17



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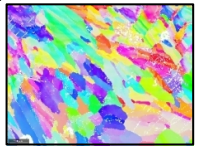
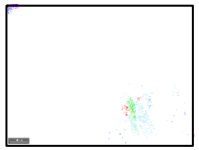


AGS – 10.31 µm
AAR – 1.69

19



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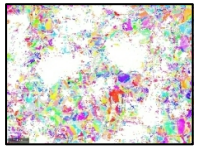
AGS – 4.03 µm
AAR – 1.21

21



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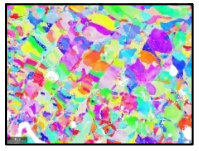
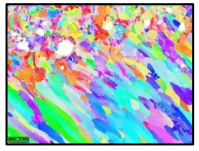


AGS – 4.45 µm
AAR – 1.57

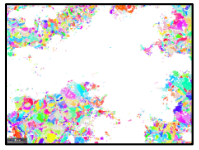
23

24



AGS – 9.71 µm
AAR – 1.48

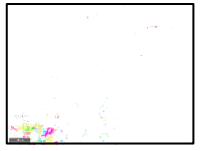
25



26

AGS – 5.13 µm
AAR – 1.70

27



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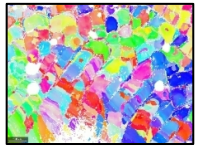
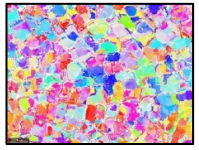


AGS – 4.06 µm
AAR – 1.34

29



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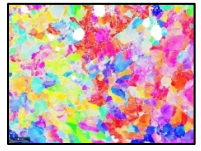
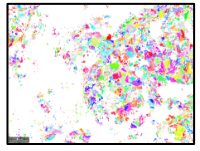


AGS – 7.96 µm
AAR – 1.88

31

32



AGS – 8.62 µm
AAR – 1.74

AGS – 22.94 µm
AAR – 1.85

AGS – 14.88 µm
AAR – 1.49

AGS – 10.37 µm
AAR – 1.98

AGS – 11.21 µm
AAR – 2.13

AGS – 13.94 µm
AAR – 1.91

AGS – 10.91 µm
AAR – 1.70

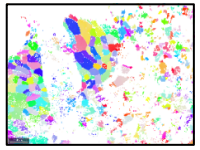
AGS – 15.86 µm
AAR – 1.66

AGS – 4.65 µm
AAR – 1.59

AGS = Average Grain Size ; AAR = Average Aspect Ratio

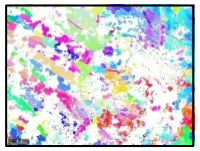
**All maps x-ipfs, FCC

1



AGS – 6.95 µm
AAR – 1.50

2



AGS – 7.23 µm
AAR – 1.45

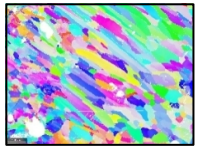
3

AGS – 22.29 µm
AAR – 1.50

4

AGS – 9.31 µm
AAR – 1.80

5

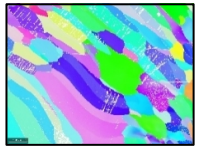


AGS – 16.51 µm
AAR – 2.20

6

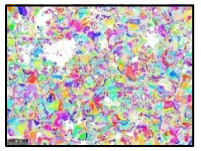
AGS – 6.37 µm
AAR – 1.77

7



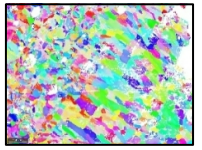
AGS – 28.63 µm
AAR – 2.01

8



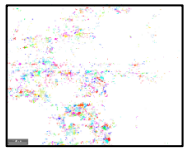
AGS – 4.90 µm
AAR – 1.84

9



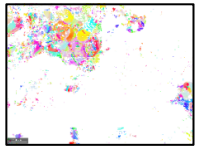
AGS – 7.98 µm
AAR – 1.74

10



AGS – 3.39 µm
AAR – 1.41

11



AGS – 4.75 µm
AAR – 1.50

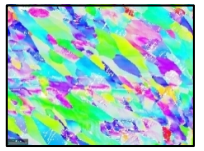
12

AGS – 19.28 µm
AAR – 1.88

13

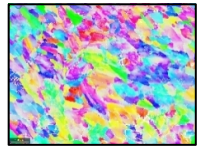
AGS – 15.11 µm
AAR – 1.60

14



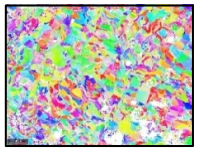
AGS – 22.45 µm
AAR – 1.82

15



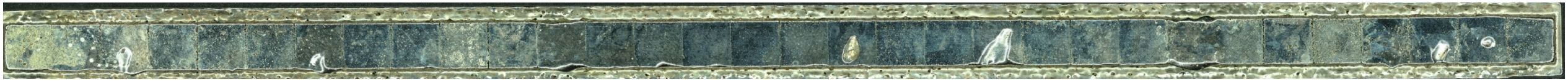
AGS – 16.54 µm
AAR – 1.94

16



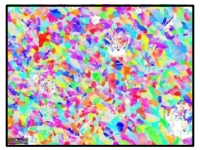
AGS – 7.23 µm
AAR – 1.90

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16



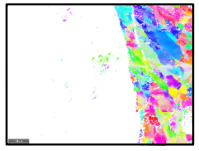
17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32

17



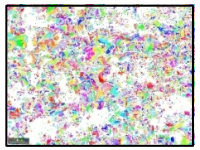
AGS – 10.65 µm
AAR – 1.66

18



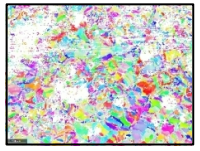
AGS – 12.80 µm
AAR – 1.49

19



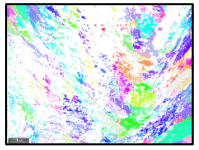
AGS – 4.21 µm
AAR – 1.61

20



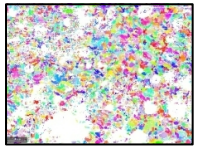
AGS – 5.18 µm
AAR – 1.67

21



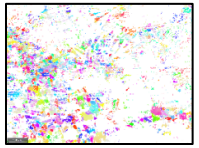
AGS – 5.39 µm
AAR – 1.44

22



AGS – 4.84 µm
AAR – 1.55

23

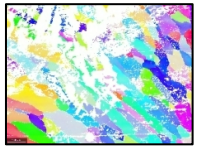


AGS – 4.61 µm
AAR – 1.45

24

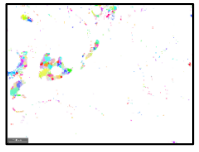
AGS – 3.26 µm
AAR – 1.37

25



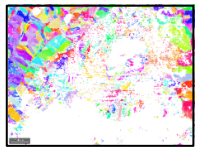
AGS – 8.87µm
AAR – 1.52

26



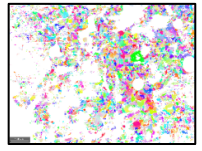
AGS – 5.09 µm
AAR – 1.42

27



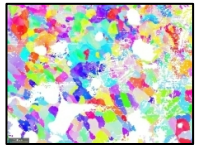
AGS – 5.61 µm
AAR – 1.48

28



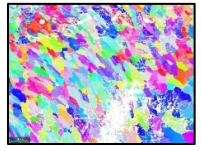
AGS – 4.40 µm
AAR – 1.52

29



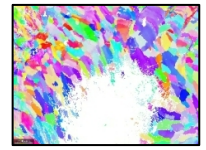
AGS – 10.97 µm
AAR – 1.58

30



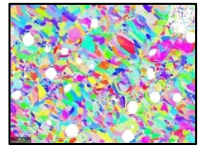
AGS – 10.84 µm
AAR – 1.51

31



AGS – 11.27 µm
AAR – 1.47

32

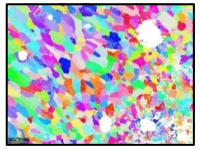


AGS – 7.71 µm
AAR – 1.99

AGS = Average Grain Size ; AAR = Average Aspect Ratio

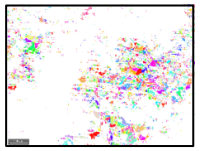
**All maps x-ipfs, FCC

1



2

AGS – 12.58 µm
AAR – 1.66

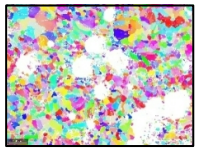


AGS – 4.16 µm
AAR – 1.47

3

AGS – 15.03 µm
AAR – 1.49

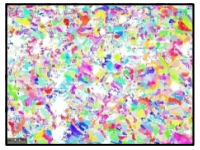
4



AGS – 8.35 µm
AAR – 1.51

5

AGS – 5.97 µm
AAR – 1.59



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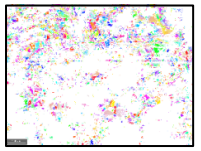
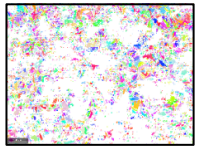


AGS – 3.90 µm
AAR – 1.43

7

AGS – 4.01 µm
AAR – 1.51



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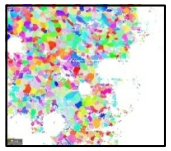
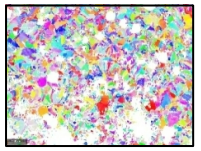


AGS – 8.95 µm
AAR – 1.40

9

AGS – 11.54 µm
AAR – 1.39

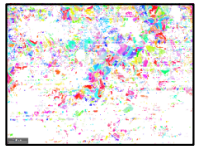
10



AGS – 5.61 µm
AAR – 1.67

11

AGS – 4.33 µm
AAR – 1.48



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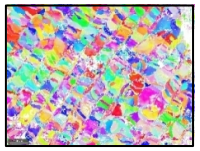
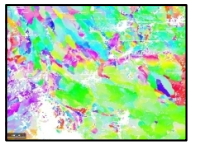


AGS – 7.86 µm
AAR – 1.82

13

AGS – 5.56 µm
AAR – 1.61

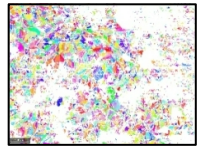
14



AGS – 9.14 µm
AAR – 1.65

15

AGS – 4.38 µm
AAR – 1.53



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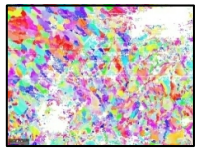
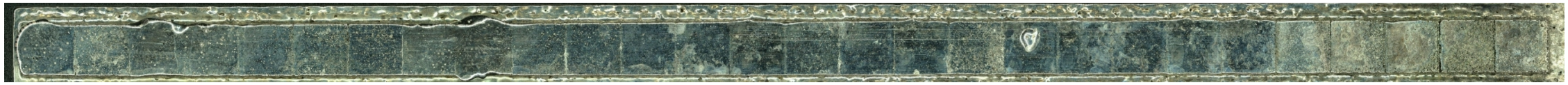


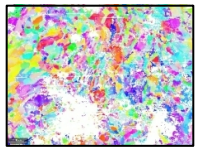
AGS – 6.42 µm
AAR – 1.64

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16



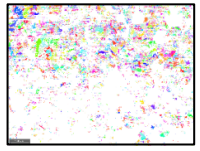
17 18 19 20 21 22 23 24 25 26 27 28

17



AGS – 6.64 µm
AAR – 1.59

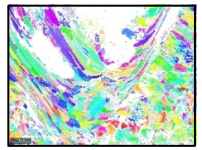
18



AGS – 3.79 µm
AAR – 1.40

19

AGS – 7.20 µm
AAR – 1.61



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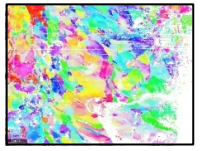
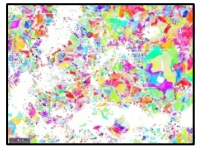


AGS – 8.84 µm
AAR – 1.48

21

AGS – 5.46 µm
AAR – 1.59



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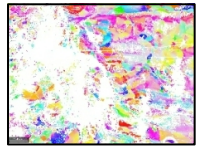
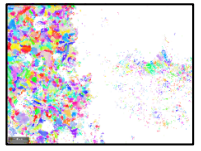


AGS – 6.02 µm
AAR – 1.42

23

AGS – 6.09 µm
AAR – 1.48

24

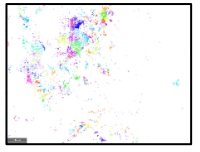


AGS – 5.49 µm
AAR – 1.43

25

AGS – 3.49 µm
AAR – 1.31

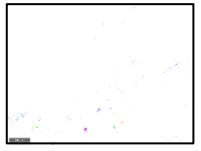
26



AGS – 3.98 µm
AAR – 1.34

27

AGS – 2.97 µm
AAR – 1.24



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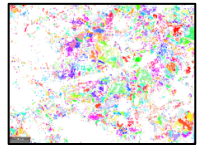

AGS – 4.29 µm
AAR – 1.42

Additional Descriptive Information Available:

- # grains indexed
- % sample area indexed
- BCC phase data
- BCC/FCC 3D phase maps
- Crystallographic orientation 3D heat maps
- Strain information (RI of grain color)

EBSD Map Data 25 - EBSD Data Map_001.ctf - Notepad

File Edit Format View Help

```
Channel Text File
Prj
Author
JobMode Grid
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YCells  496
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AcqE1   0.0000
AcqE2   0.0000
AcqE3   0.0000
Euler angles refer to Sample Coordinate system (CS
Phases  2
2.866;2.866;2.866       90.000;90.000;90.000    Ir
3.660;3.660;3.660       90.000;90.000;90.000    Ir
Phase   X       Y       Bands   Error   Euler1  Eu
2       0.0000  0.0000  7       0       47.0262 39
2       1.5000  0.0000  7       0       47.3745 39
2       3.0000  0.0000  7       0       45.2229 39
0       4.5000  0.0000  0       3       0.0000  0.
2       6.0000  0.0000  7       0       44.9578 39
0       7.5000  0.0000  0       3       0.0000  0.
0       9.0000  0.0000  0       3       0.0000  0.
2       10.5000 0.0000  8       0       45.0037 38
2       12.0000 0.0000  7       0       44.9420 39
2       13.5000 0.0000  7       0       45.3626 39
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2       16.5000 0.0000  7       0       44.9056 39
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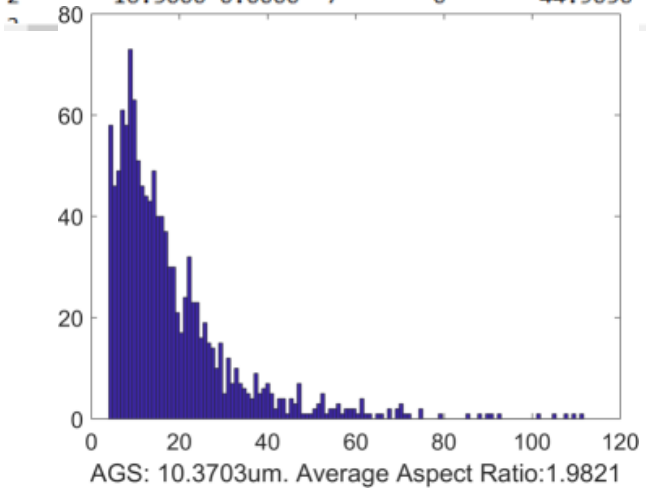
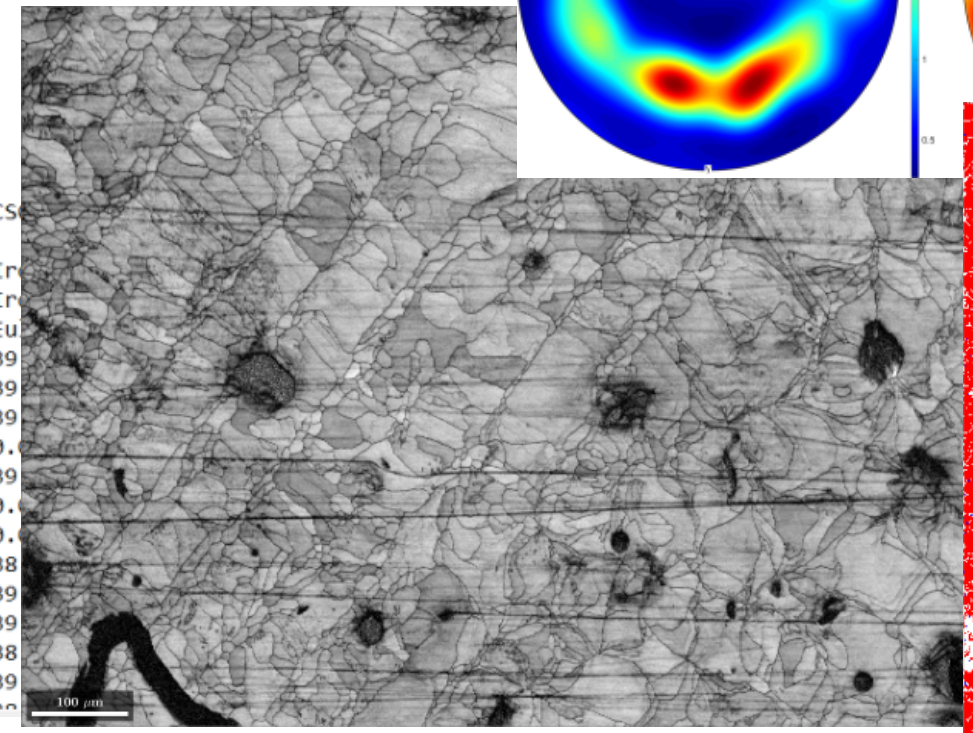
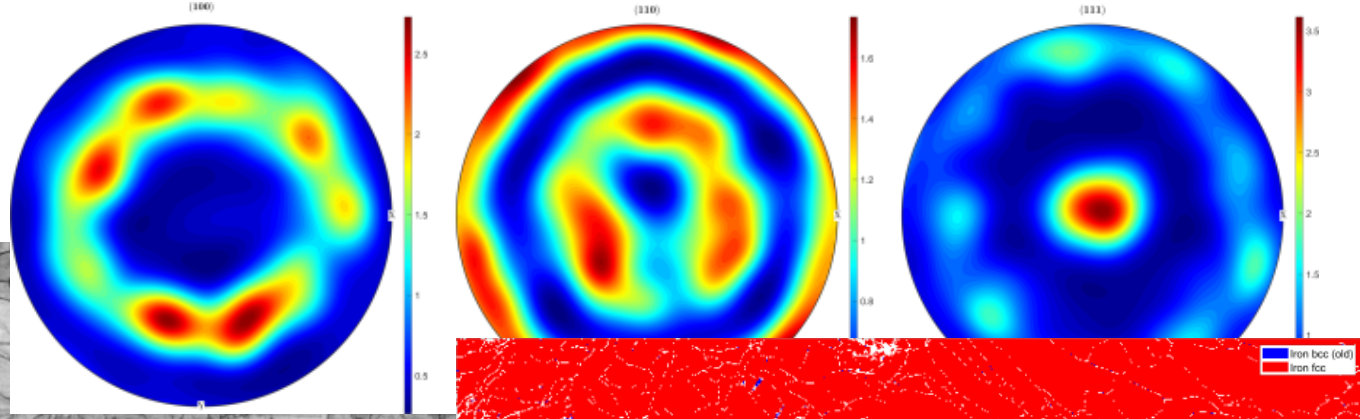
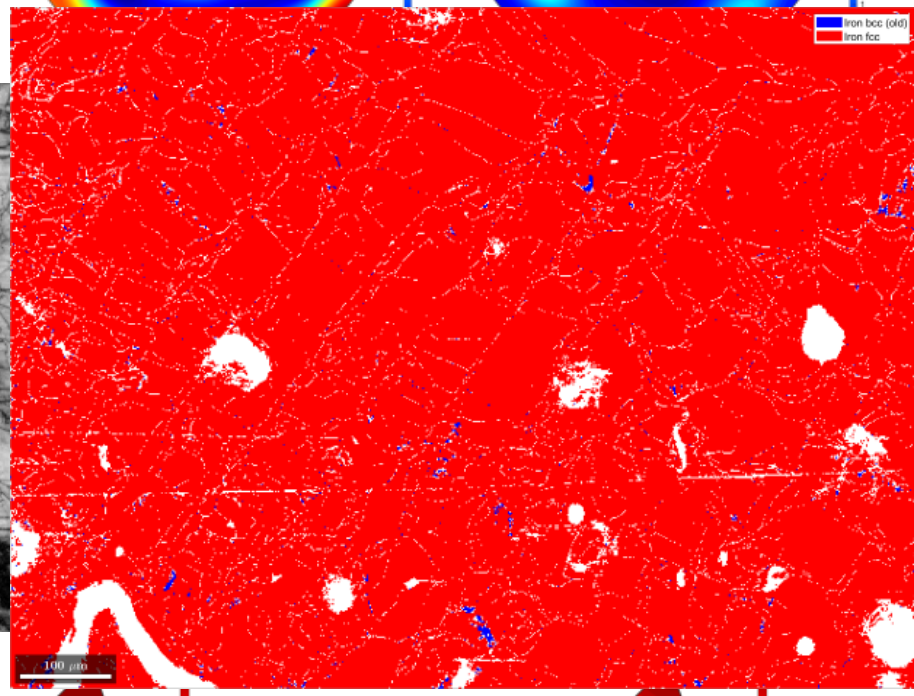
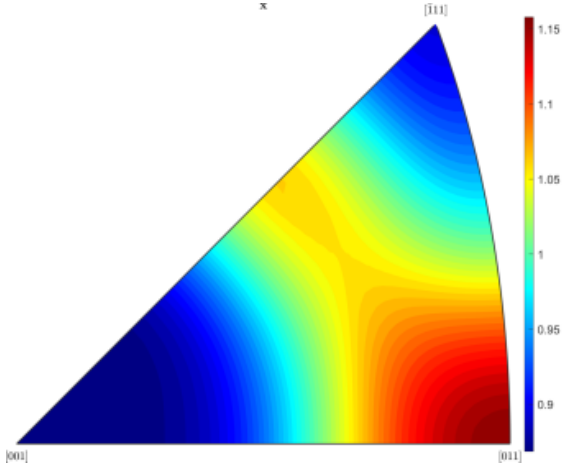
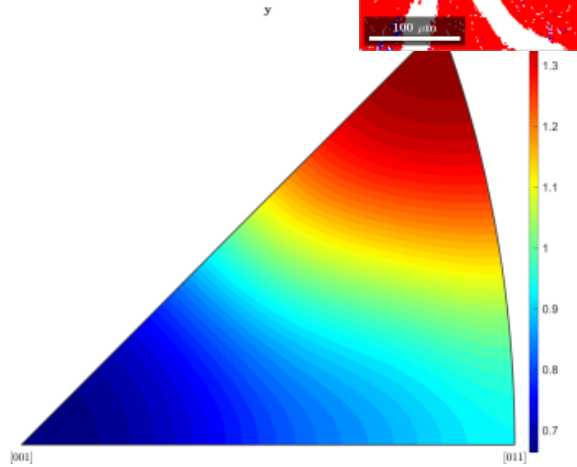


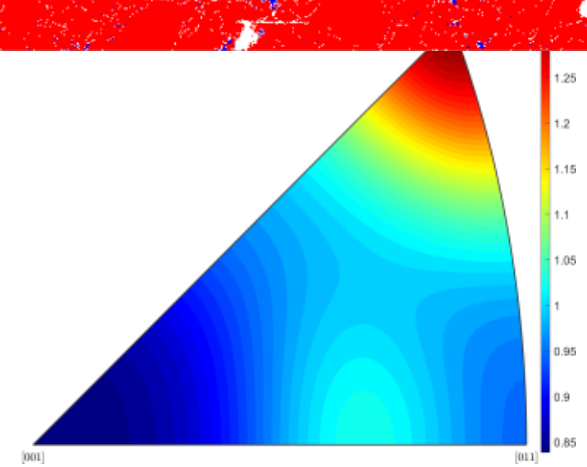




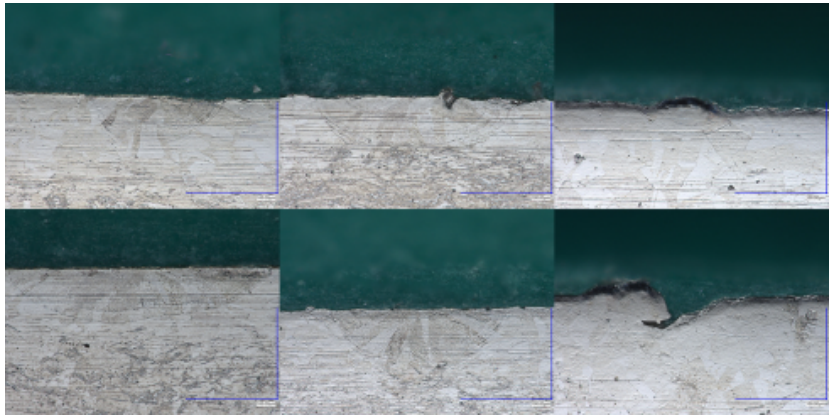






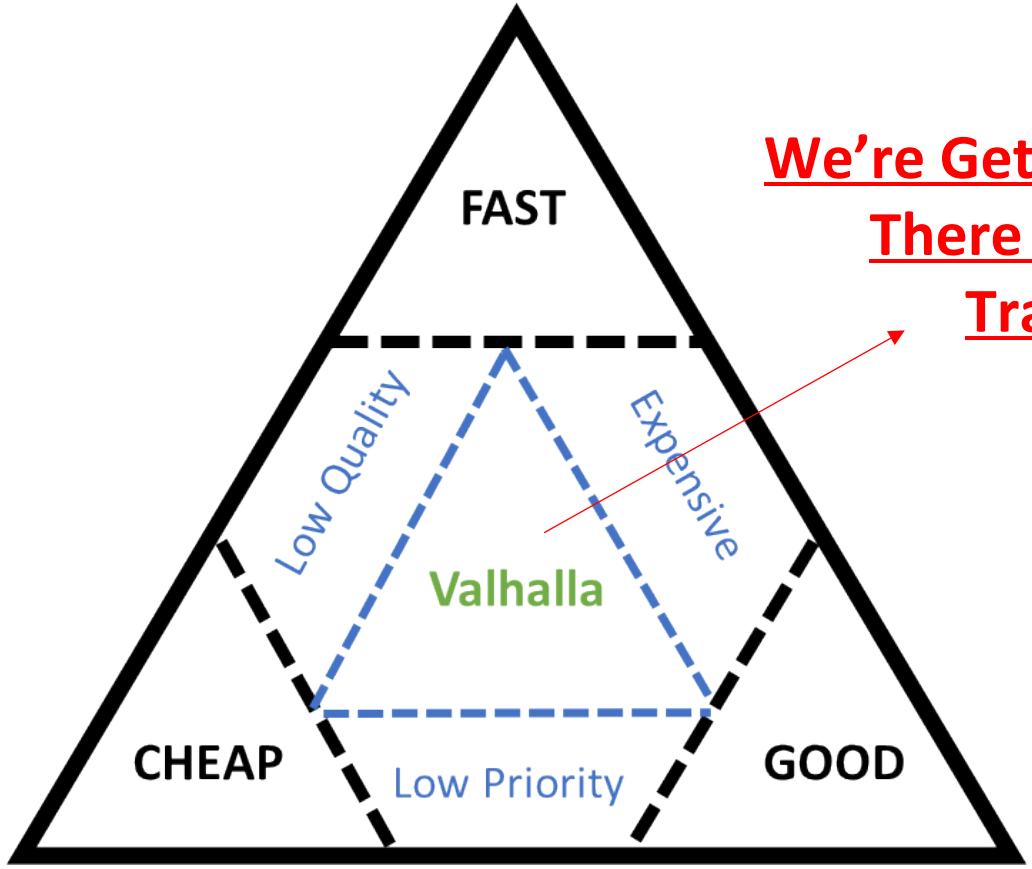


CONCLUSIONS



<u>High-Throughput Melt Pool Imaging</u>
~16x Improvement in Prep Time

<u>High-Throughput EBSD</u>
~32x Improvement in Prep Time



<u>We're Getting Closer, But There Are Always Tradeoffs!</u>

Questions?

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