

Electropolishing 6061 Al Targets

SAND2020-1728PE

Metal Micromachining Team



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Chemistry for Electropolishing

■ Electropolishing:

- 60% Phosphoric acid
- 40% Polyethylene glycol – 1000 monomer chains
- T=50C – 80C
- Stainless steel counter electrode
- Additives tried:
 - ◆ Glycolic acid
 - ◆ Thiodene

■ Barrier anodization process:

- Ammonium tartrate
- Stainless steel counter electrode
- T = RT
- Etchant for oxide: 0.4M chromic acid and 0.2M phosphoric acid



Example of Polishing Runs

	voltage (V)	pulse (son/soff)	on-time (s)	roughness (μm)	mass loss rate (mg/s)	rate of roughness change (μm/s)	roughness/mass loss ratio
	2		3600	1.875333333	-0.010722222	5.72685E-05	-0.005341105
	1.14		600	1.9663	-0.034666667	0.000151611	-0.004373397
	3		600	2.14575	-0.009833333	0.000299083	-0.030415254
	2	.2/.8	120	2.205666667	-0.193333333	0.000499306	-0.002582615
	2.5	.2/.8	120	2.027833333	-0.260833333	-0.001481944	0.005681576
	1.5	.2/.8	120	2.702	-0.036666667	0.005618056	-0.153219697
	2	.1/.9	60	2.718	-0.465	0.02047	-0.044021505
stirr off	2	.1/.9	60	2.593666667	-0.225	-0.002072222	0.009209877
stirr off, 60C	2	.1/.9	60	2.509166667	-0.105	-0.001408333	0.013412698
stirr off, 60C	2.5	.1/.9	60	2.8995	-0.078333333	0.006505556	-0.083049645
stirr off, 60C	2	.4/.6	10	0.803166667	-0.25	0.006583333	-0.026333333
stirr off, 60C	2.5	.4/.6	10	0.721333333	-0.04	-0.008183333	0.204583333
stirr off, 60C	2.5	.6/.4	10.2	0.878166667	-0.049019608	0.015375817	-0.313666667
stirr off, 60C	2.5	.4/1.6	10	0.880166667	-0.05	0.0002	-0.004
stirr off, 60C	2.5	.4/.2	10	0.768	-0.02	-0.011216667	0.560833333
stirr off, 60C	2	.4/.2	10	0.792	-0.05	0.0024	-0.048
stirr off, 60C	2.5	.4/.1	10	0.787333333	-0.06	-0.000466667	0.007777778
stirr off, 60C	2.5		10	0.841833333	-0.07	0.00545	-0.077857143
stirr off, 60C	2.5		600	0.837666667	-0.006333333	-6.94444E-06	0.001096491

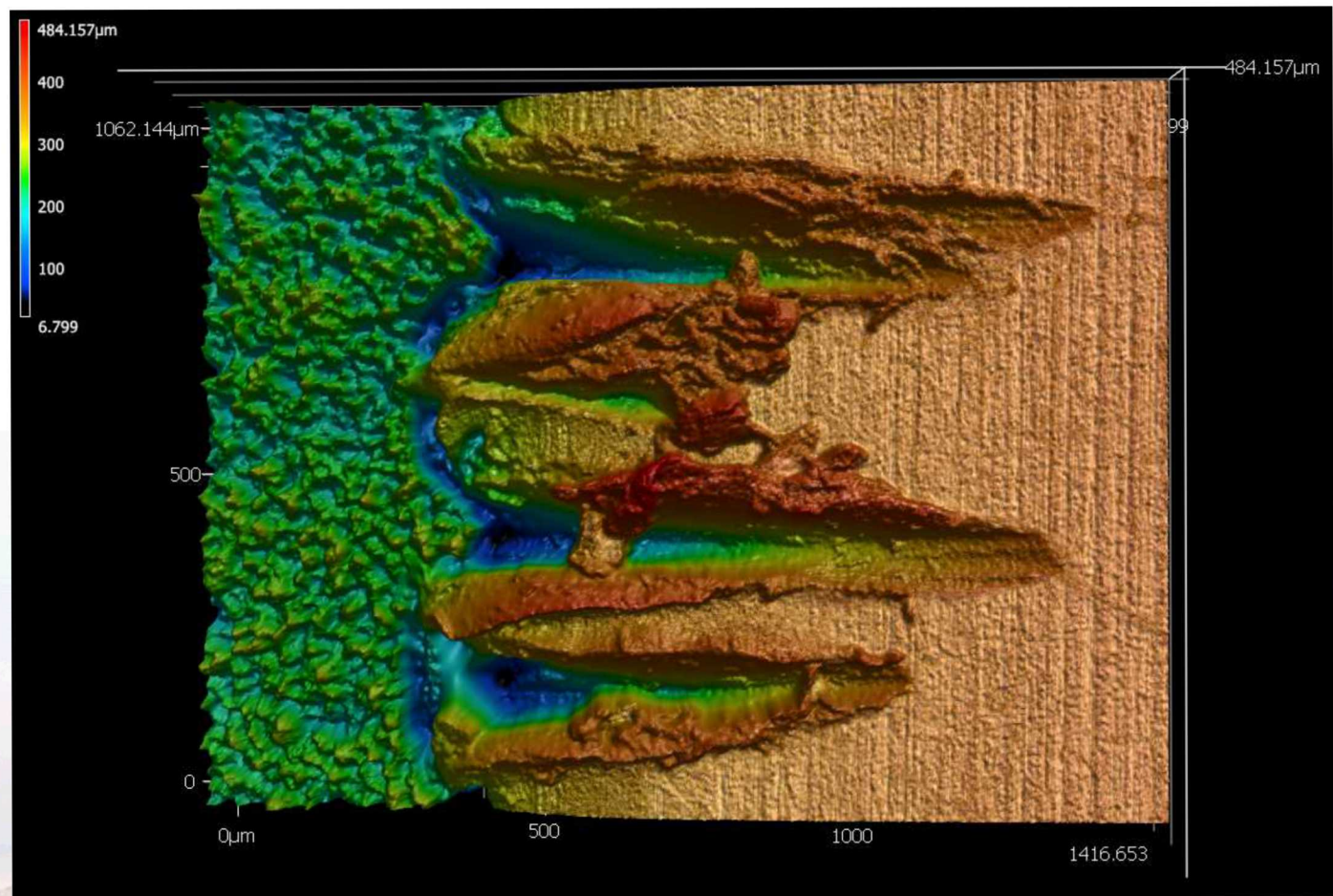


Example of Polishing Runs

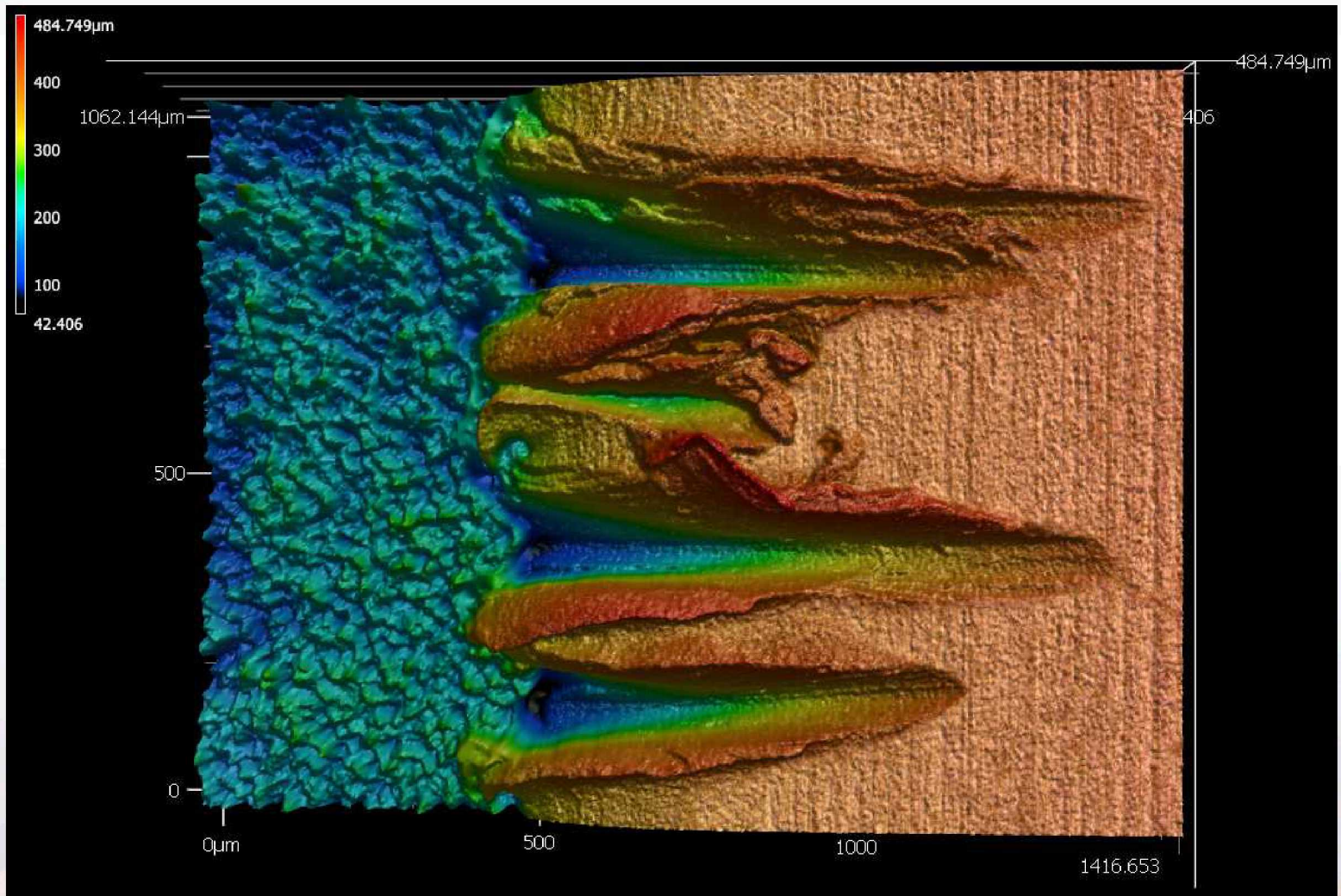
voltage	prepolish vertical roughness	prepolish horizontal roughness	avg roughness	mass	thickness	avg thickness	mass loss rate (mg/s)	thickness loss rate (mm/s)	rate of roughness change	roughness/mass loss ratio
plate 1	1.414	1.788	1.669166667	2.0257	0.486	0.483666667				
	1.21	2.253			0.48					
	1.298	2.052			0.485					
2	3600		1.875333333	1.9871	0.466	0.470666667	-0.010722222	-0.000746612	5.72685E-05	-0.005341105
	1.409	1.682			0.466					
	1.351	2.277			0.48					
1.14	1.982	2.551	1.9663	1.9663	0.454	0.455333333	-0.034666667	-0.005429651	0.000151611	-0.004373397
	600				0.451					
	1.516	1.897			0.461					
3	600		2.14575	1.9604	0.449	0.453333333	-0.009833333	-0.000732064	0.000299083	-0.030415254
	1.602	2.075			0.45					
	2.414	2.492			0.461					
2V pulse	120		2.205666667	1.9372	0.437	0.436666667	-0.193333333	-0.030637255	0.000499306	-0.002582615
	2.226	2.436			0.437					
	1.694	2.668			0.436					
reset	2.109	2.101		1.9372	0.437	0.436666667	#VALUE!	#VALUE!	#VALUE!	#VALUE!
					0.437					
					0.436					
2.5	120 (0.2s on 0.8s off)		2.027833333	1.9059	0.423	0.418	-0.260833333	-0.03562341	-0.001481944	0.005681576
	2.072	2.233			0.416					
	1.756	2.047			0.415					
1.5	120 (0.2s on 0.8s off)		2.702	1.9015	0.415	0.415333333	-0.036666667	-0.005316321	0.005618056	-0.153219697
	2.258	2.741			0.418					
	3.335	2.474			0.413					
PLATE 2 prepolish	1.516	1.481	1.4898		2.038	0.481	0.48			
	1.428	1.659				0.479				
	1.365					0.48				
2V pulse	60 (0.1s on 0.9s off)		2.718	2.0101	0.466	0.467	-0.465	-0.045138889	0.02047	-0.044021505
	3.039	2.612			0.468					
	2.208	3.025			0.467					
2V pulse	2.624	2.8	2.593666667	1.9966	0.46	0.460333333	-0.225	-0.023792529	-0.002072222	0.009209877
	60 (0.1s on 0.9 off, stirr off)				0.461					
	2.374	2.726								
2V pulse	2.507	2.742	2.509166667	1.9903	0.456	0.455666667	-0.105	-0.016895969	-0.001408333	0.013412698
	2.23	2.983			0.455					
	2.755	2.616			0.456					
2.5V pulse	60 (0.1s on 0.9 off, stirr off 60C)		2.8995	1.9856	0.456	0.486333333	-0.078333333	0.112167764	0.006505556	-0.083049645
	3.152	2.914			0.547					
	2.397	2.701			0.456					
	3.339	2.894								



Pre Polish Image

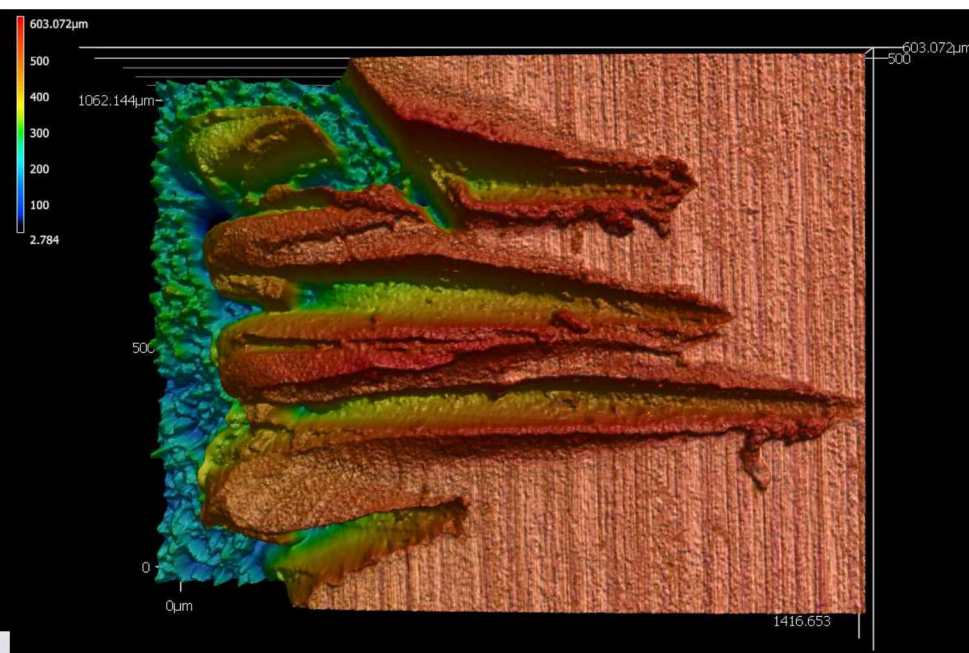
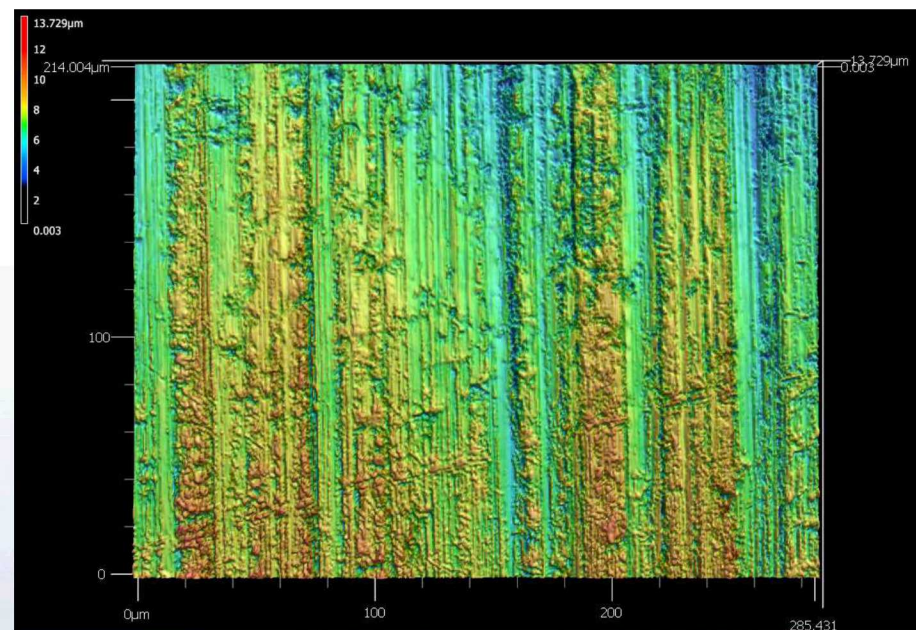


Post Polish Image



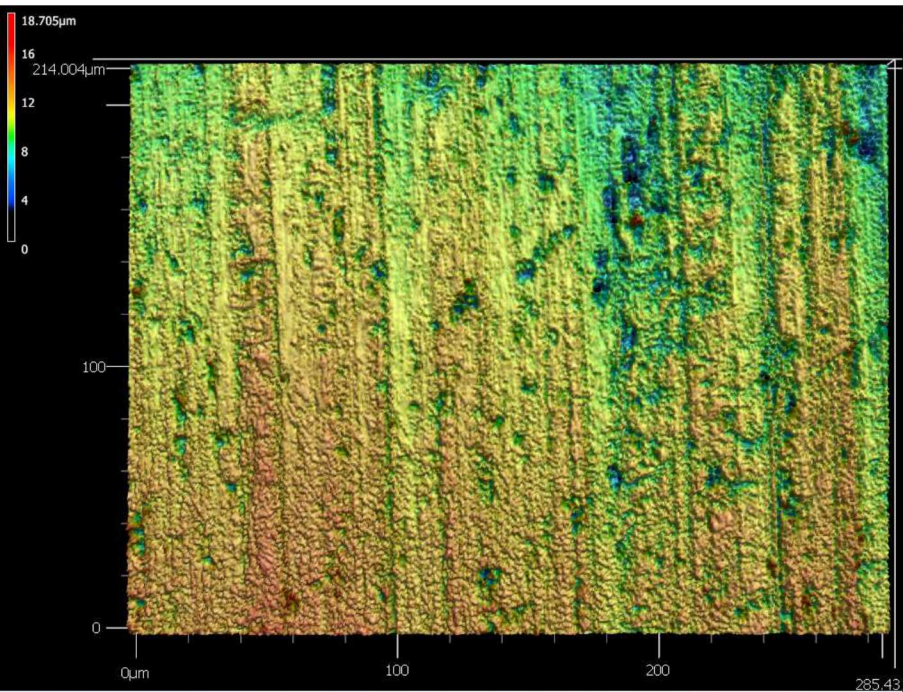


voltage (V)	pulse (son/soff)	Total on-time (s)	roughness (μm)	mass loss rate (mg/s)	rate of roughness change ($\mu\text{m/s}$)	roughness/mass loss ratio
prepolish			0.693333333			





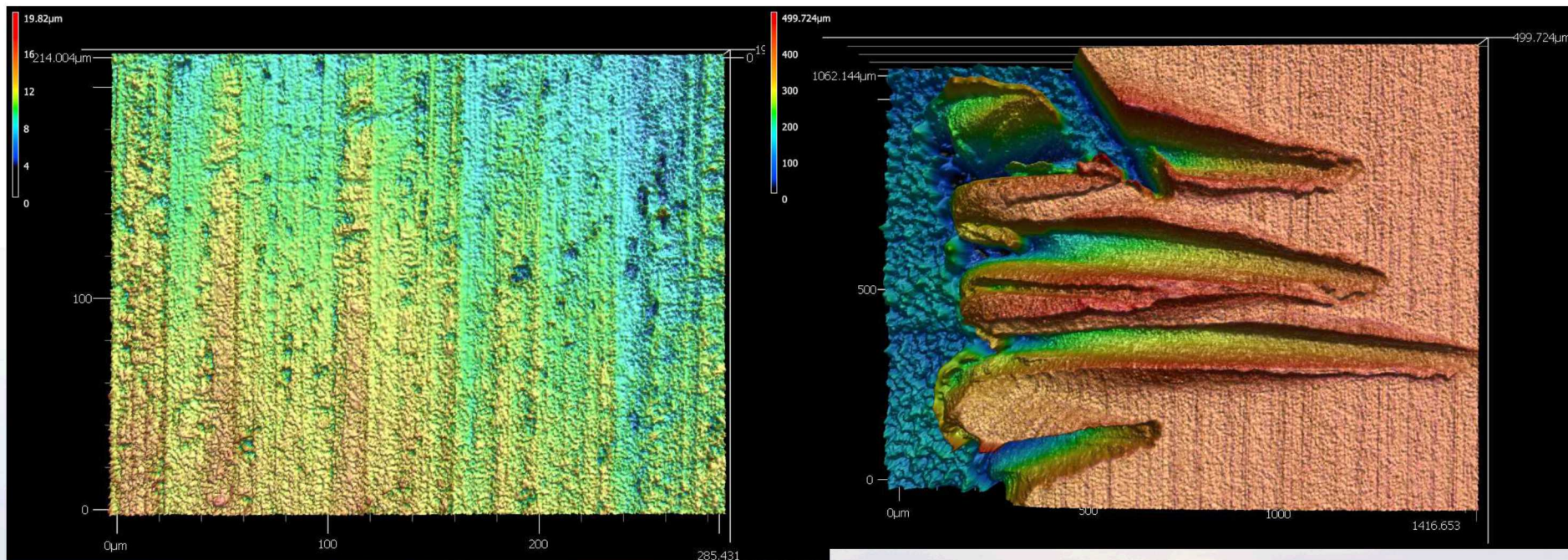
voltage (V)	pulse (son/soff)	Total on-time (s)	roughness (μm)	mass loss rate (mg/s)	rate of roughness change ($\mu\text{m/s}$)	roughness/mass loss ratio
NaOH		20	0.881			





voltage (V)	pulse (son/soff)	Total on-time (s)	roughness (μm)	mass loss rate (mg/s)	rate of roughness change ($\mu\text{m/s}$)	roughness/mass loss ratio
2.5	.4/.2	10	0.7845	-0.03	-0.005433333	0.181111111

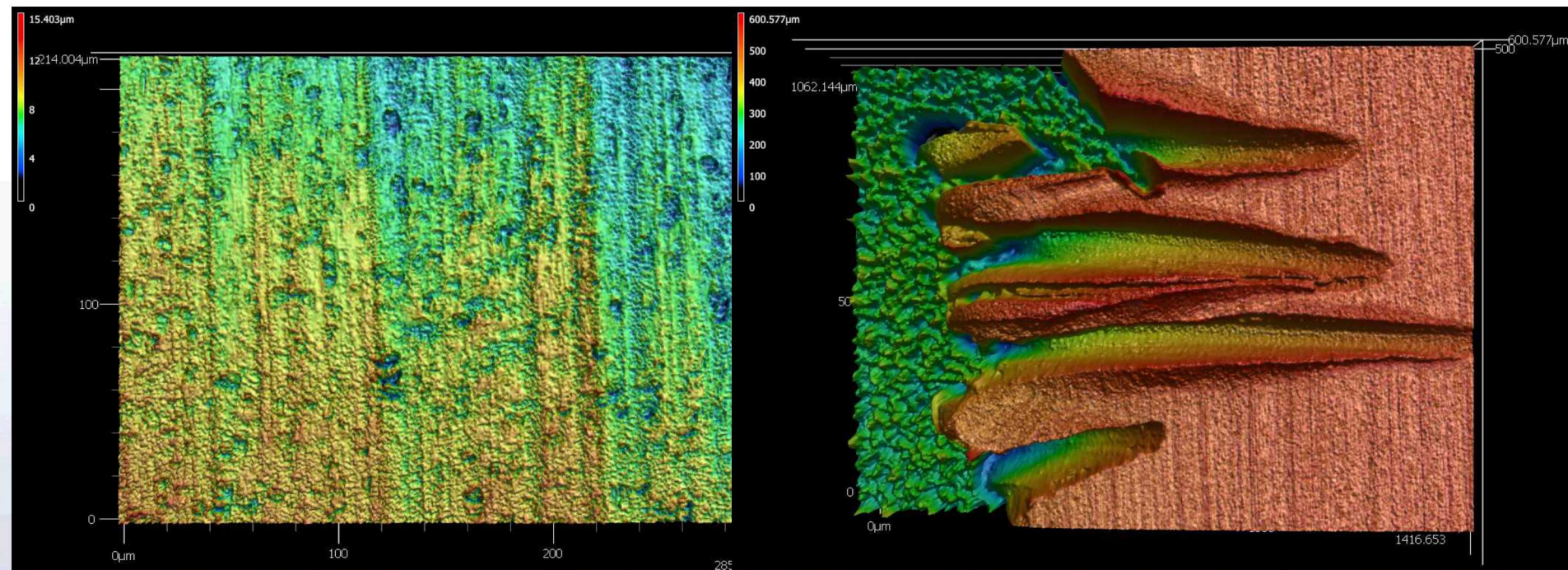
Stirring off, 50C, 10mM Thiodene





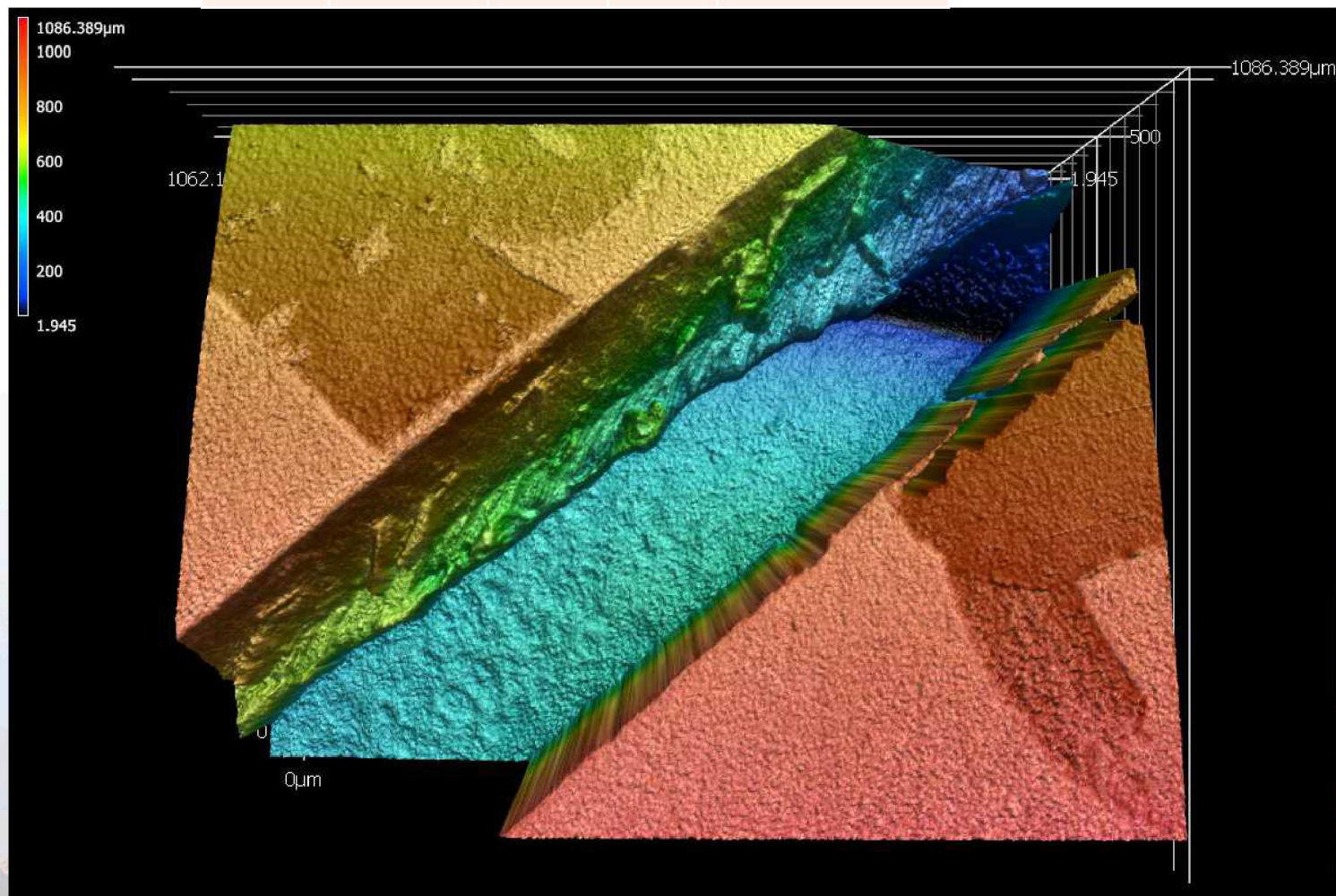
voltage (V)	pulse (son/soff)	Total on-time (s)	roughness (μm)	mass loss rate (mg/s)	rate of roughness change ($\mu\text{m/s}$)	roughness/mass loss ratio
2.35	.4/.2	10	0.7556667	-0.001069	-0.004466667	4.178359838

Stirring off, 50C, 15mM Thiodene



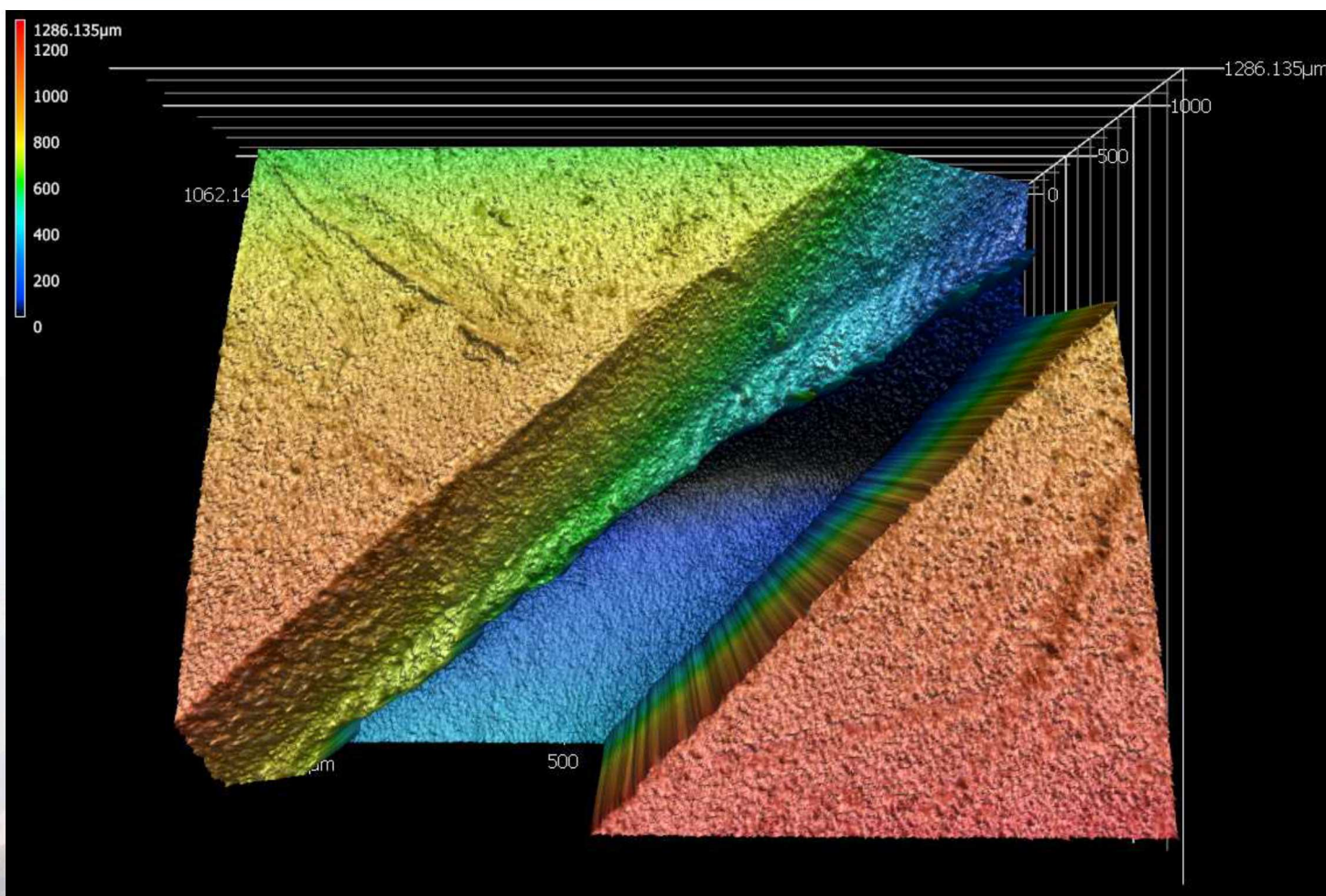


voltage (V)	pulse (son/soff)	Total on-time (s)	roughness (μm)	Conditions
prepolish			2.021	





voltage (V)	pulse (son/soff)	Total on-time (s)	roughness (μm)	Conditions
2.35	0.2/0.8	30	3.2	stirr 300rpm, 90C





Path Forward

- **Work on barrier anodization process**
- **“Mixed” pulse and DC process**
- **Other chemistries:**
 - 18% perchloric acid, 18% glycerol and 64% ethanol, at a voltage of 20 V and at room temperature⁸
 - 820 ml H_3PO_4 + 160 gr CrO_3 + 135 ml H_2SO_4 + 100 ml water, using Al cathode in 50 voltage for 5 min.
 - ♦ etch in 25 ml HNO_3 + 75 ml distilled water for 30-60 sec.





Electropolishing 6061 Al for Gabe S.

- **Chemistry 1:**

- ♦ 18% perchloric acid
- ♦ 18% glycerol
- ♦ 64% ethanol
- ♦ voltage of 20 V and T = room temperature

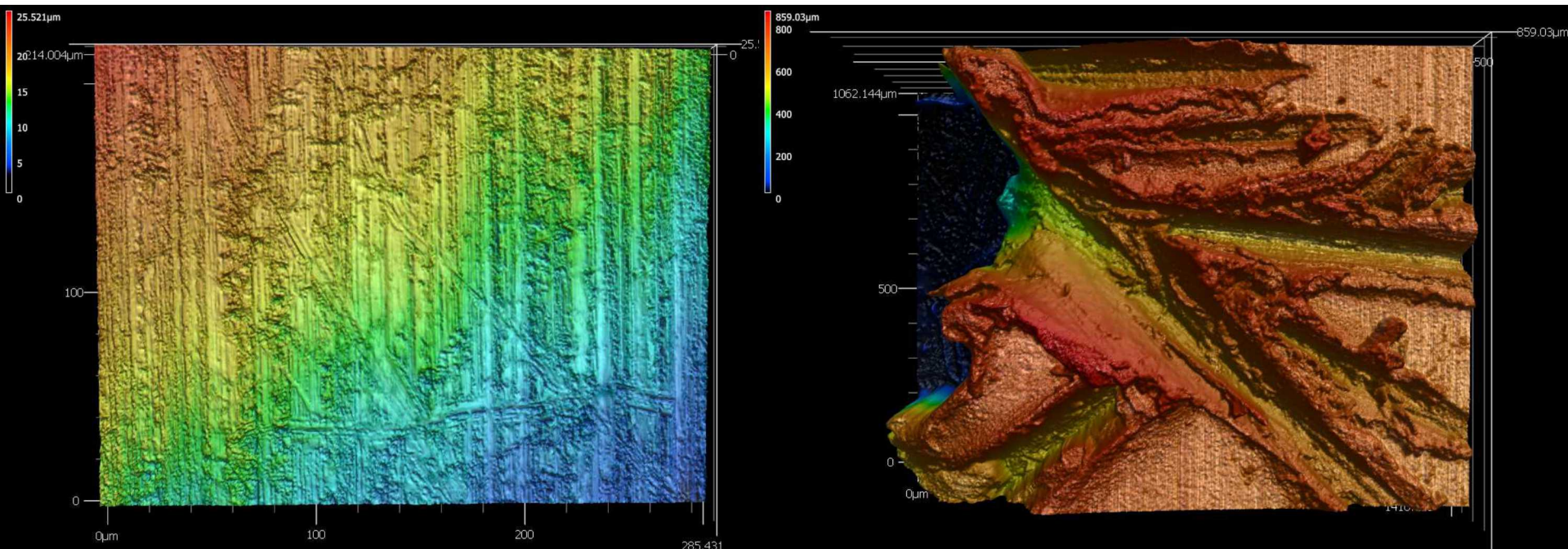
- **Chemistry 2:**

- ♦ 82 ml H_3PO_4
- ♦ 16 gr CrO_3
- ♦ 13.5 ml H_2SO_4
- ♦ 10 ml water
- ♦ Al cathode at 50 voltage for 5 min.
- ♦ etch in 25 ml HNO_3 + 75 ml distilled water for 30-60 sec.



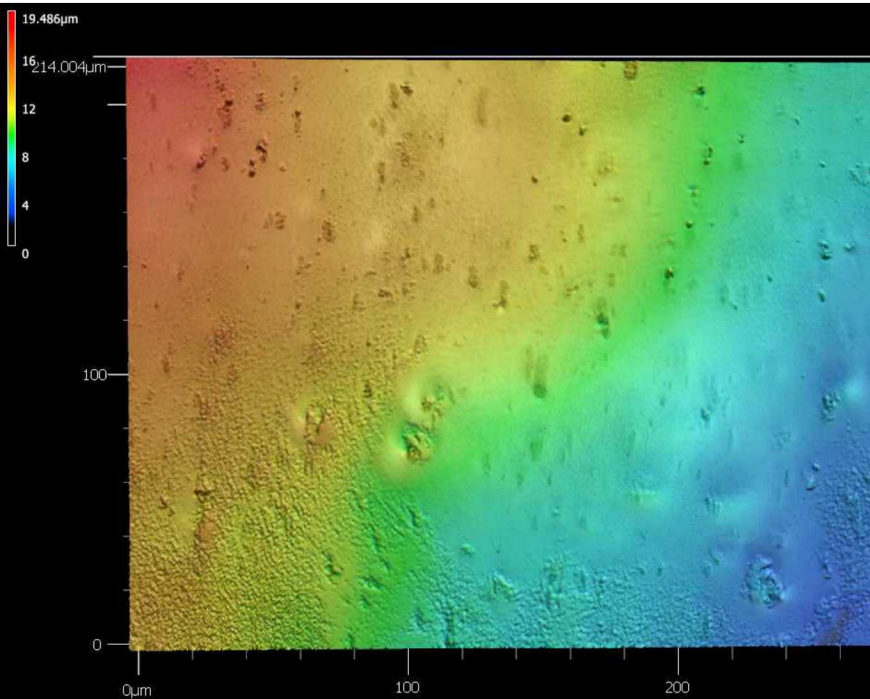
Prepolish Test Image

Voltage (V)	On-time	avg rough (μm)	Mass (g)	%mass loss rate	change in rough rate	rough/mass loss
Prepolish		0.594333	0.6744			



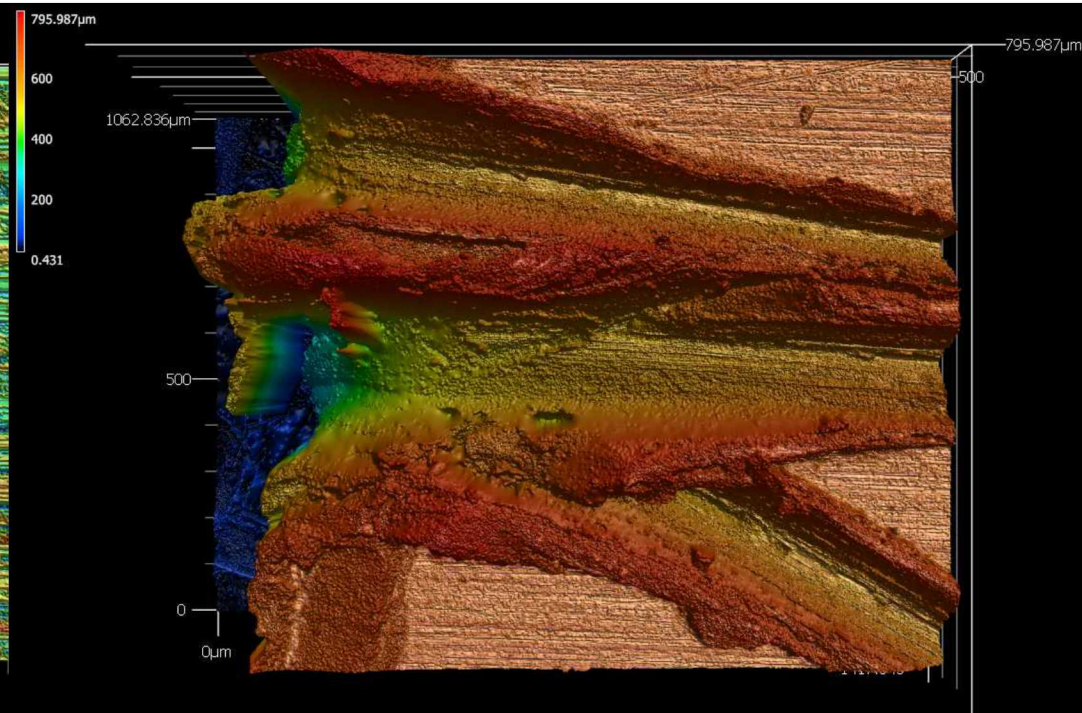
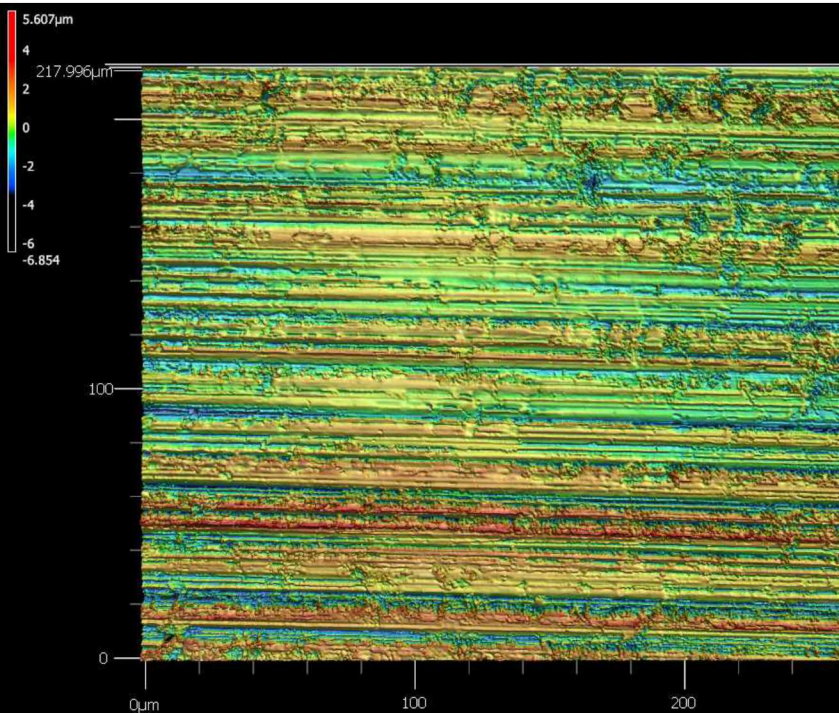
Best Seen Result (Operations >50V Require Training)

Voltage (V)	On-time	avg rough (μm)	Mass (g)	%mass loss rate	change in rough rate	rough/mass loss
60	30	0.305167	0.6432	-0.001542112	-0.009638889	6.250448718



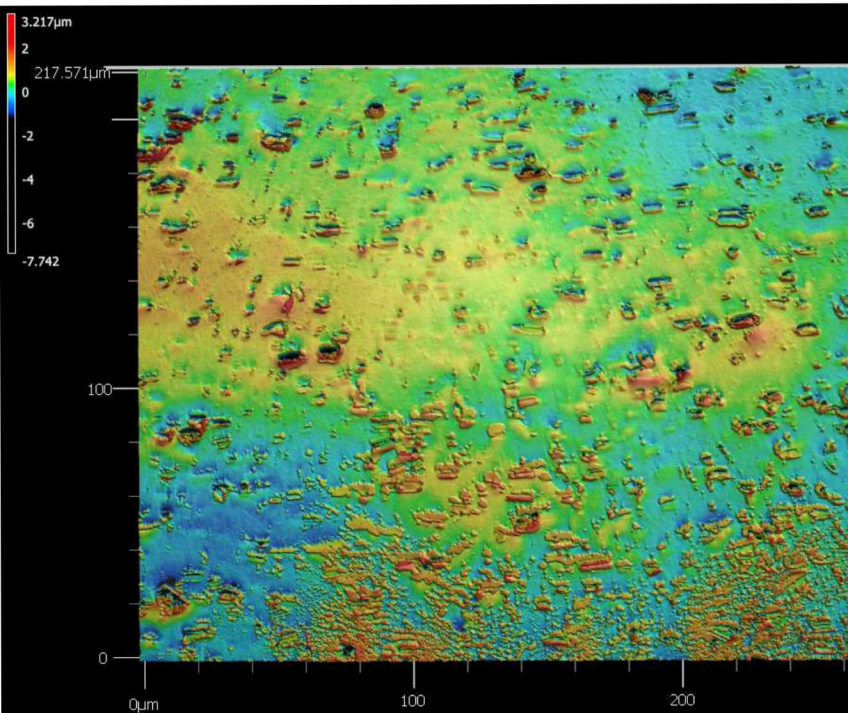
Prepolish Test Image

Voltage (V)	On-time	avg rough (μm)	Mass (g)	%mass loss rate	change in rough rate	rough/mass loss
Prepolish		0.851	1.0124			



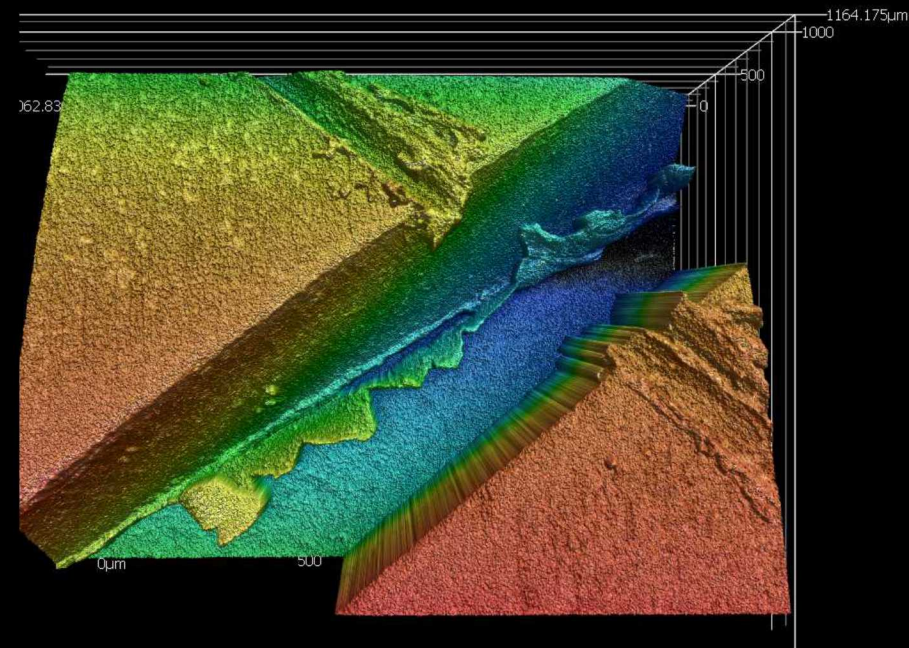
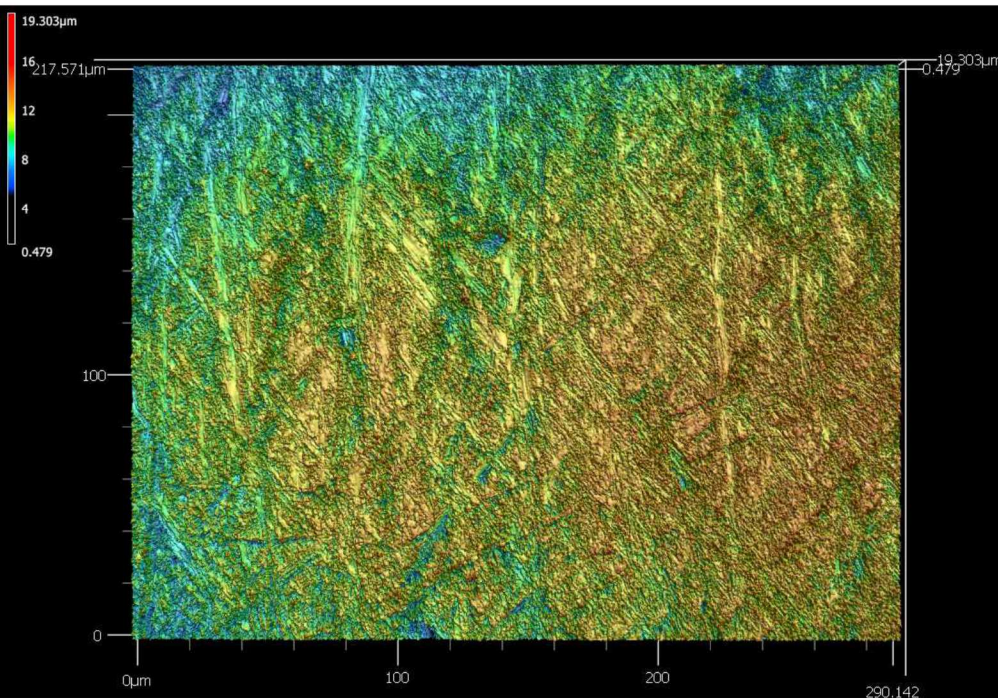
Best Parameters: 3s on, 3s off, high agitation, distant electrodes, 11A limit

Voltage (V)	On-time	avg rough (μm)	Mass (g)	%mass loss rate	change in rough rate	rough/mass loss
50	15	0.359	0.7554	-0.001619	-0.0088	5.4358723



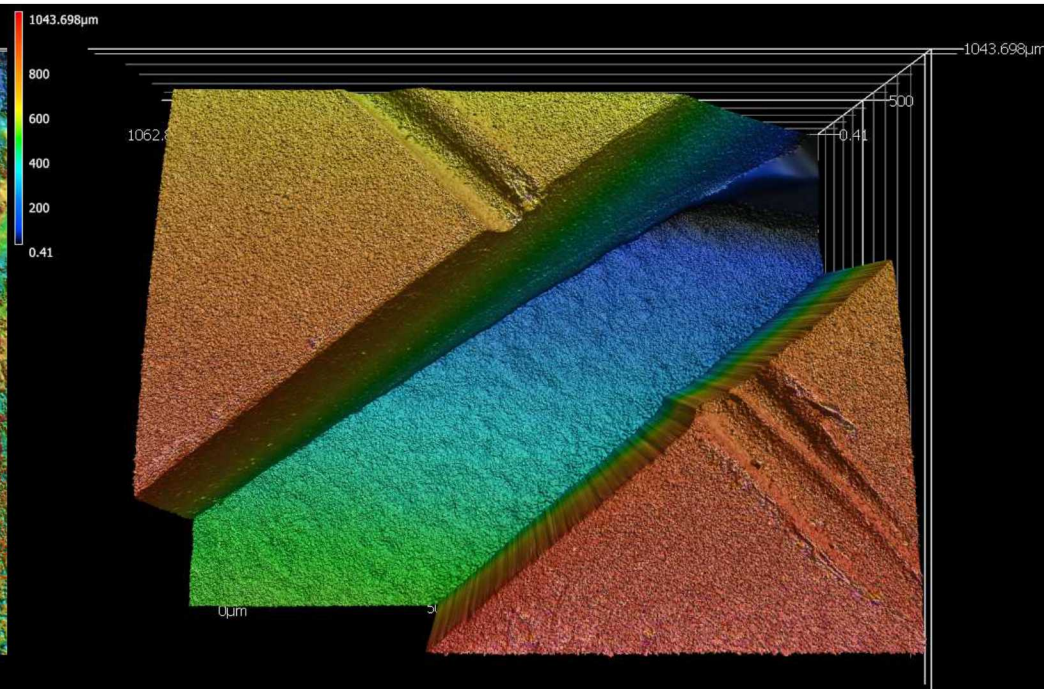
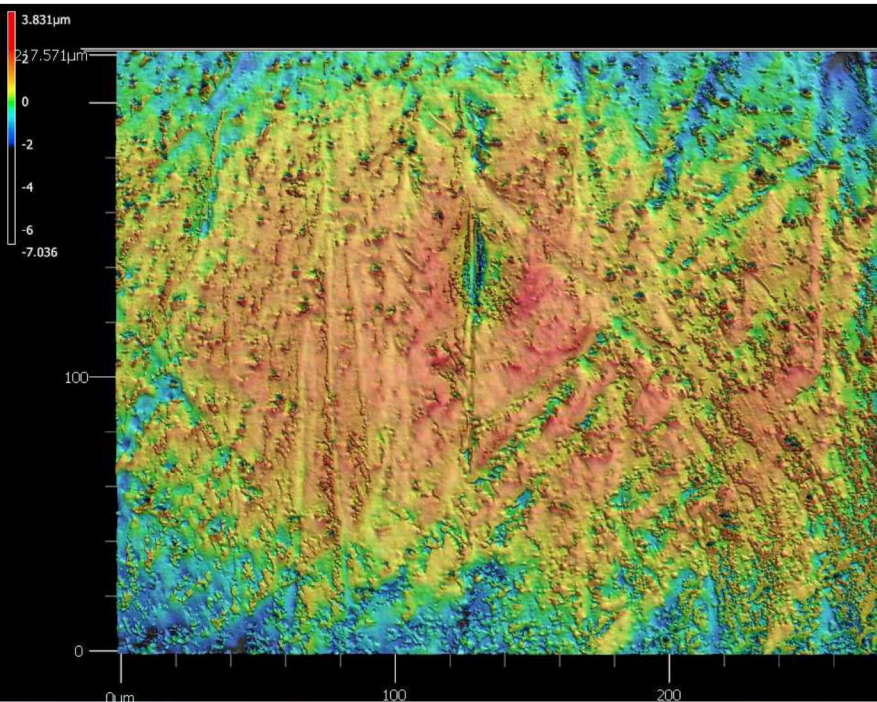
Prepolish: 500-micron target

Voltage (V)	max current (A)	time on (s)	mass (g)	% mass loss rate	thickness (mm)	roughness (μm)
prepolish			0.4706		0.50	0.967

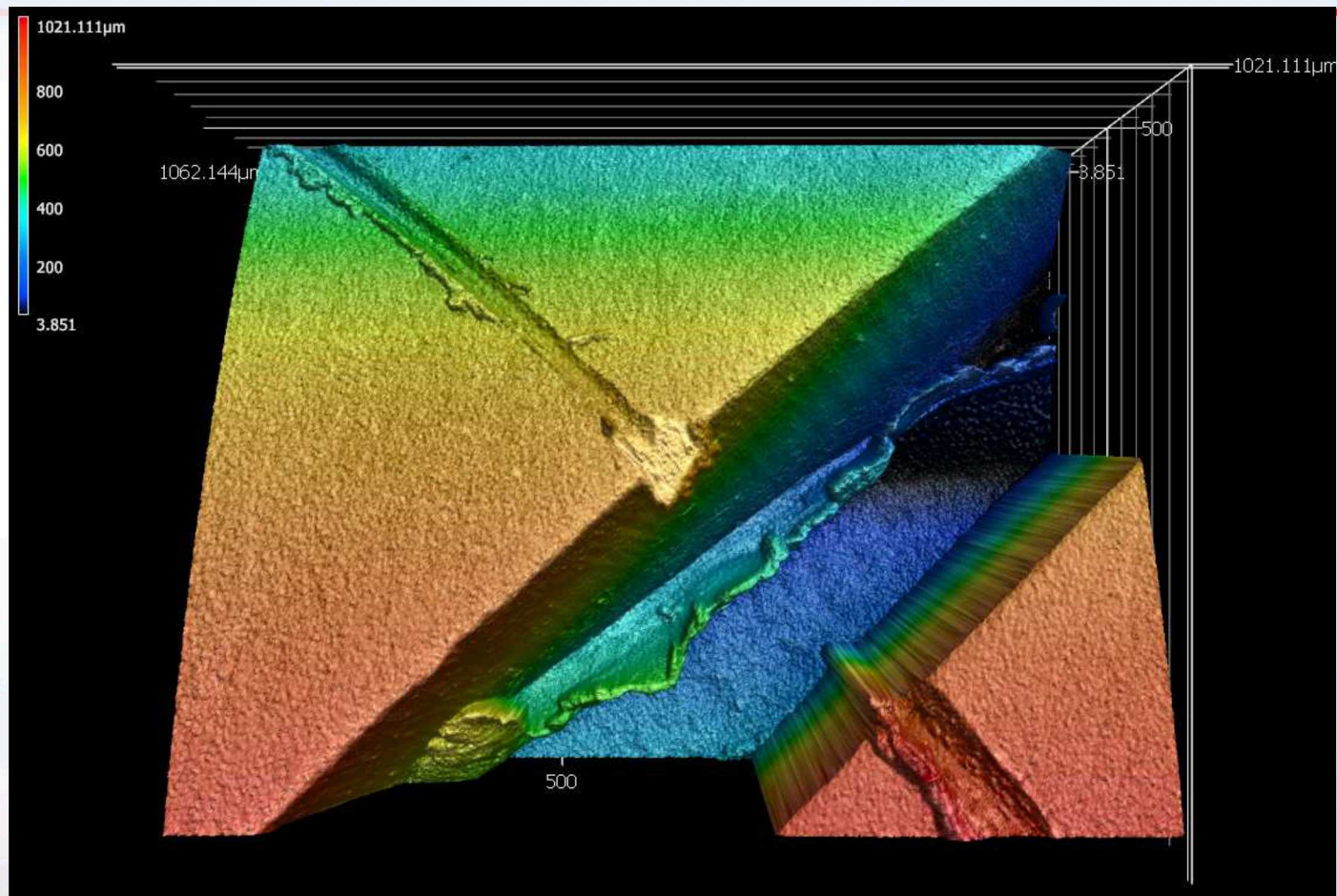


Post-Polish Using Chosen Parameters

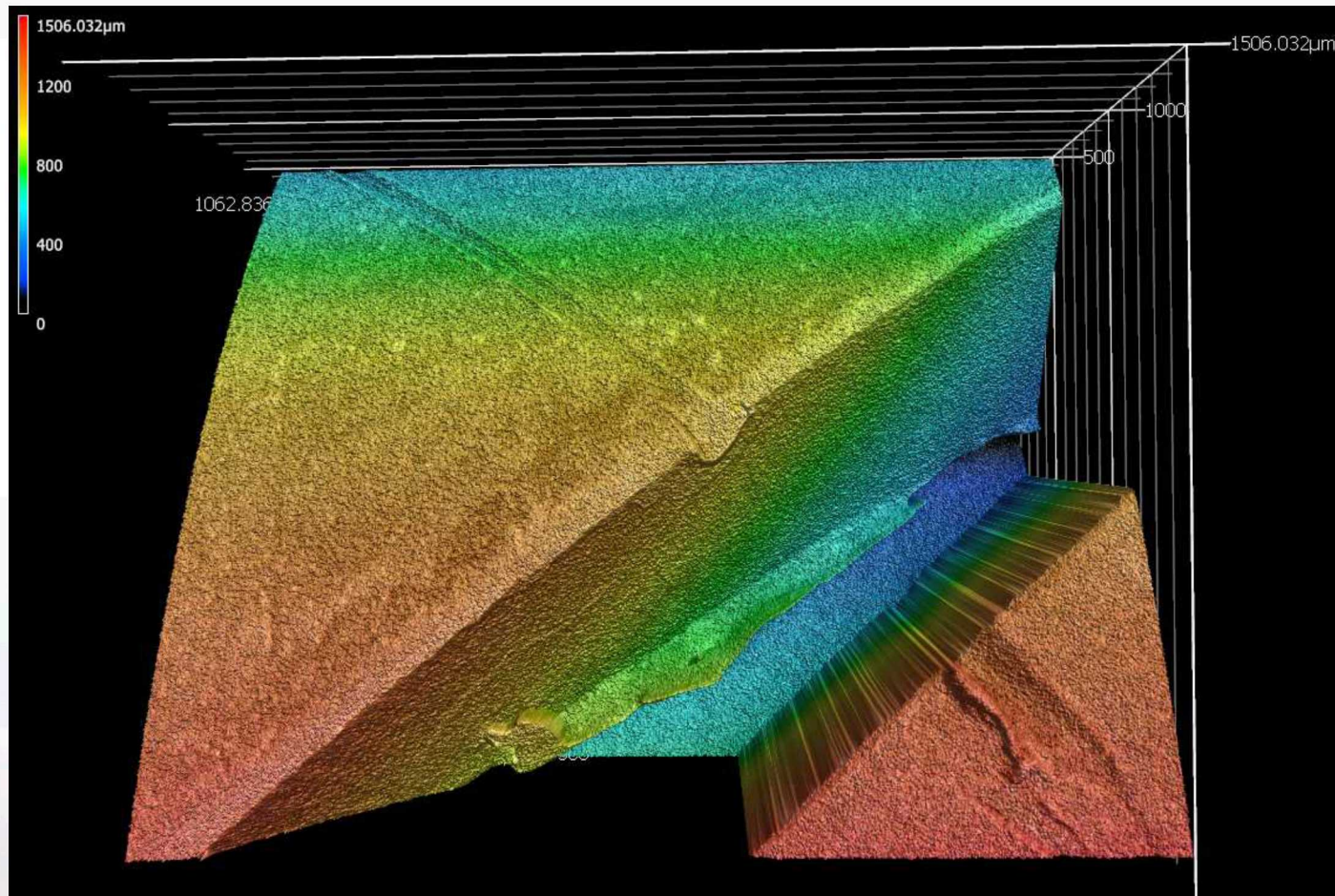
Voltage (V)	max current (A)	time on (s)	mass (g)	% mass loss rate	thickness (mm)	roughness (μm)
50	11.55	45	0.4532	-0.0014308	0.50	0.531



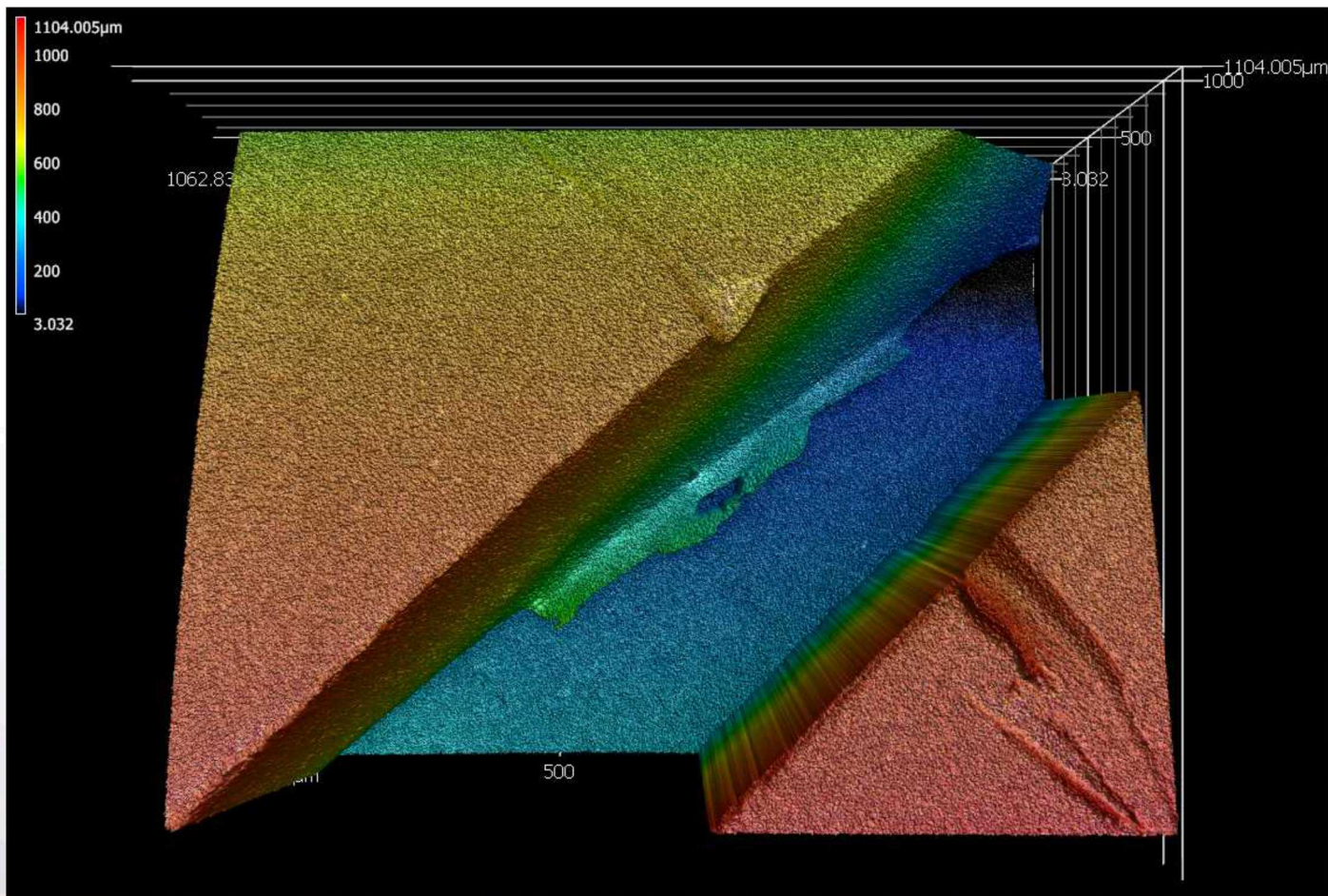
Prepolish: 500 micron part



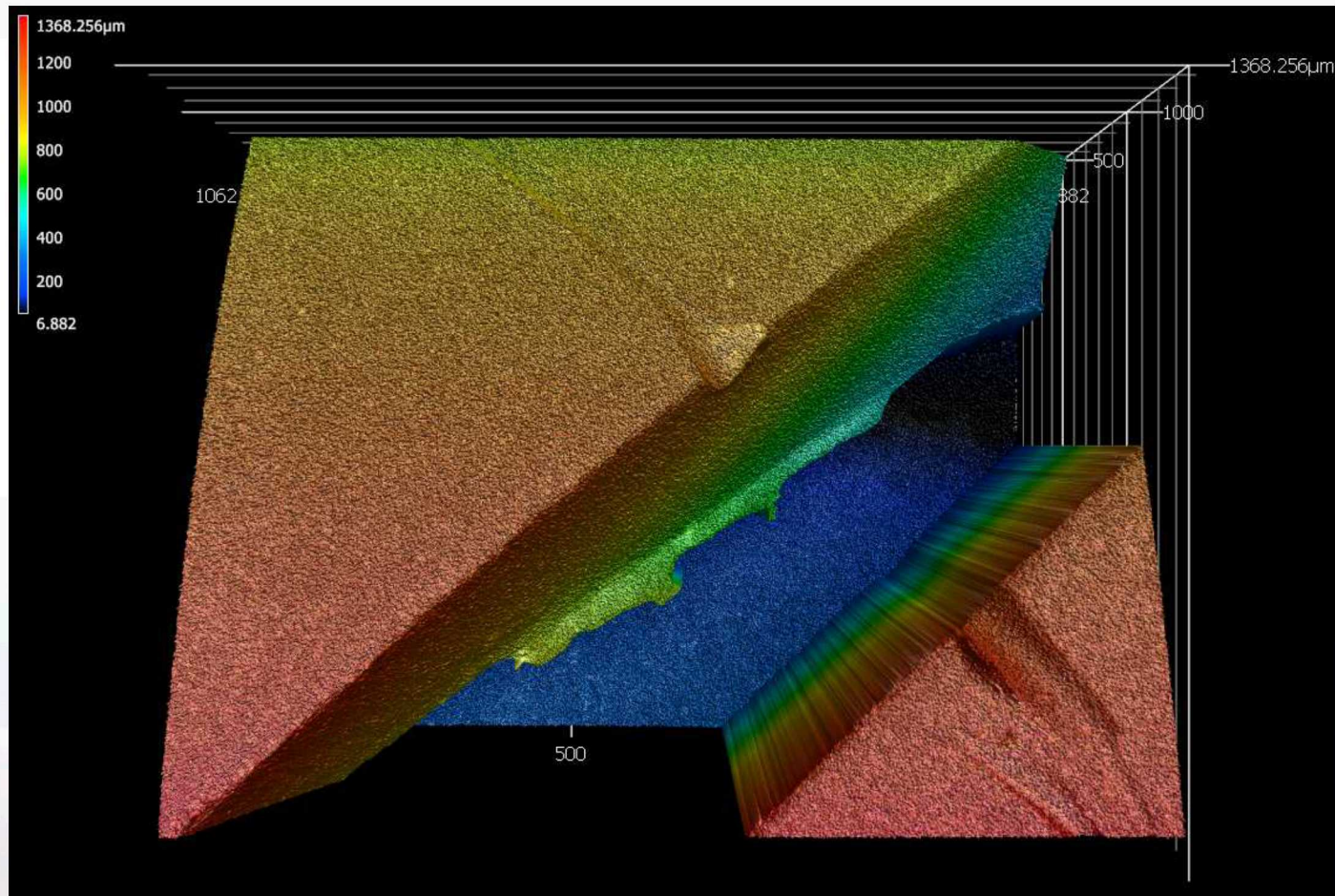
15s total on-time



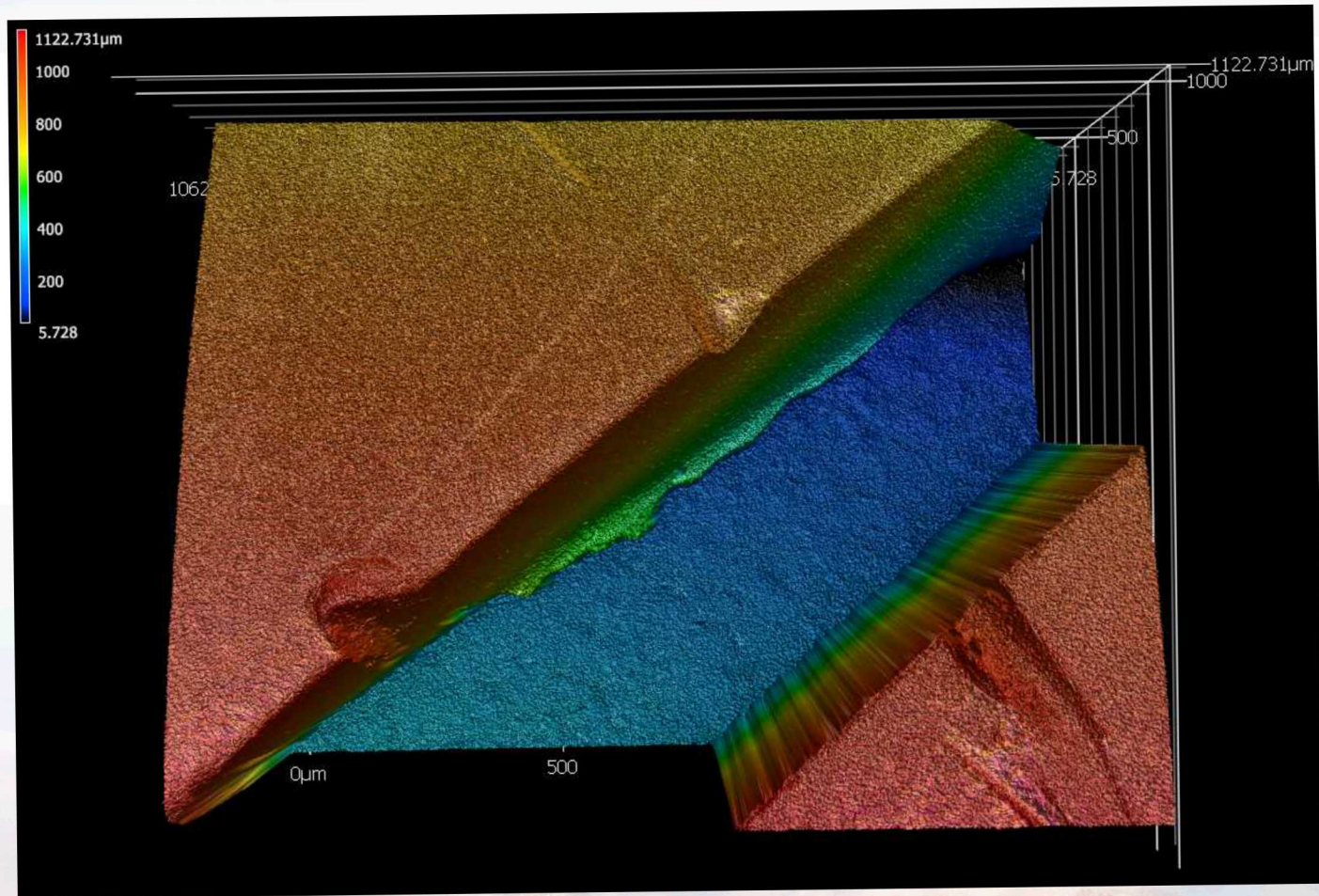
30s total on-time



45s total on-time

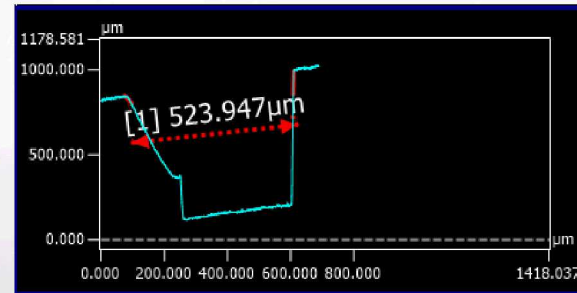
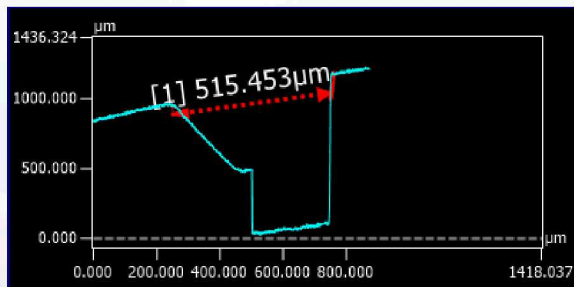
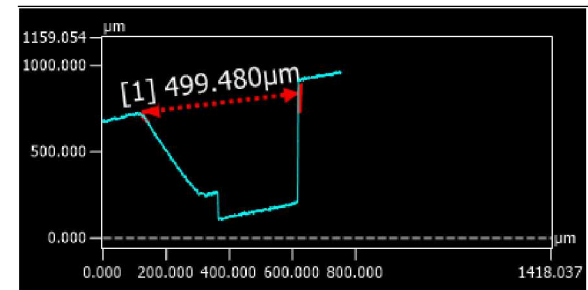
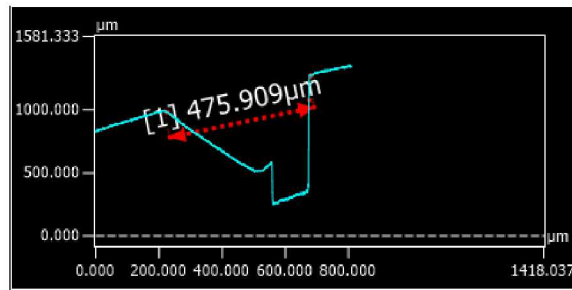
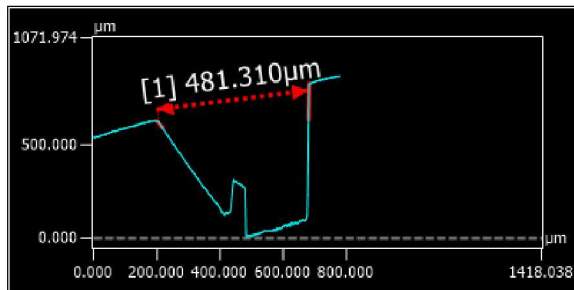


60s total on-time

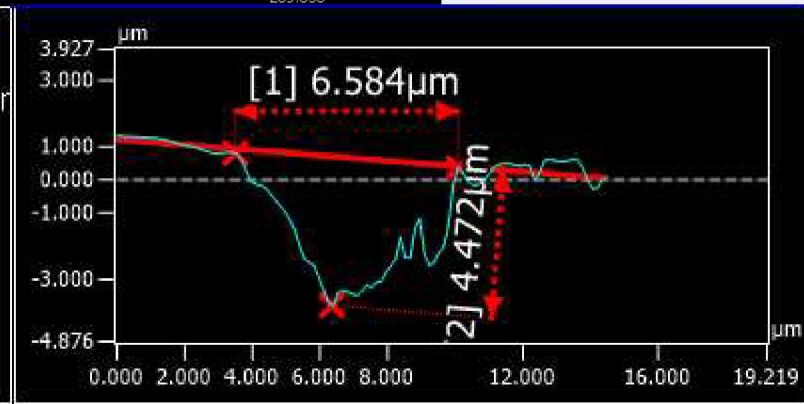
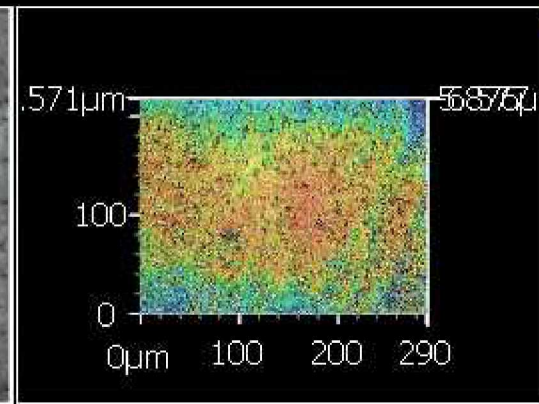
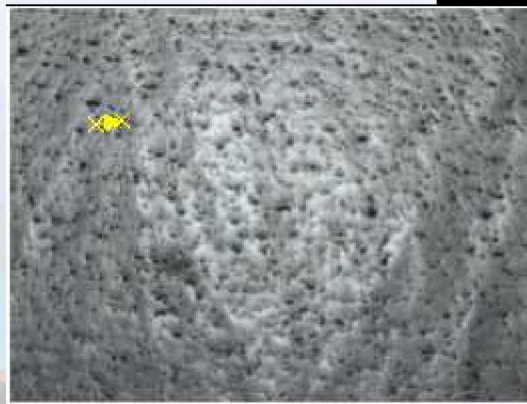
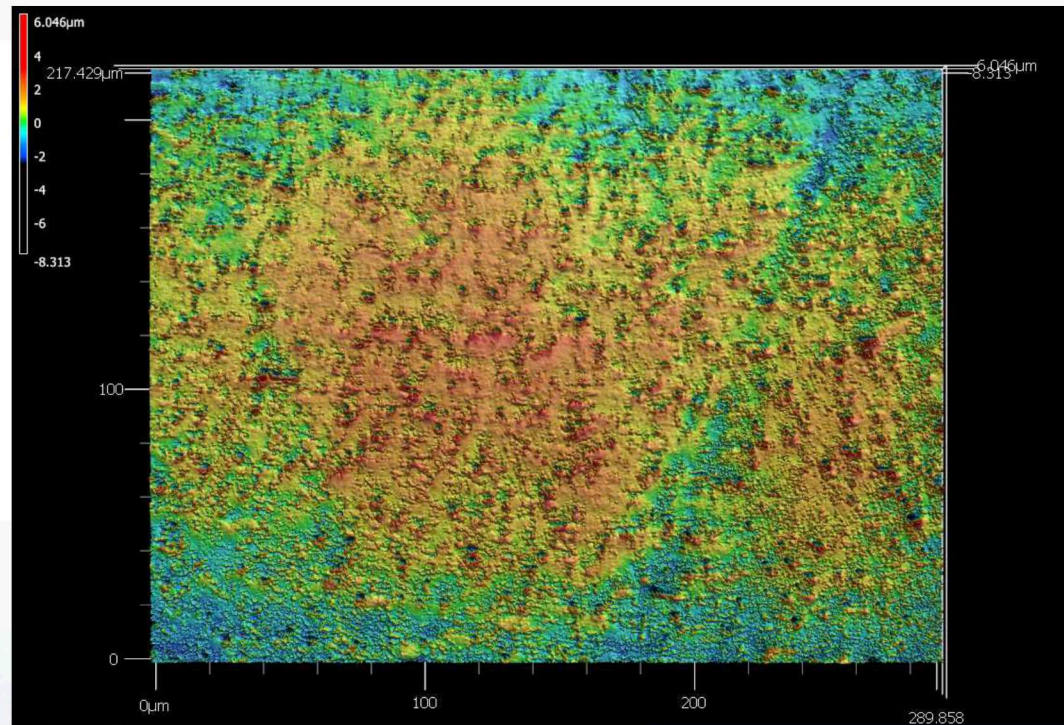


Gap Progression: 50V 11A in 15s intervals

■ ~ 10 μm / 15 s



Pit Size





Chemistry 1 vs 2

■ Chem 1

- Hazardous
- Good burr-removal
- Sensitive to agitation
 - ◆ Inconsistent surface roughness (300-1000 nm)

■ Chem 2

- Relatively safe (no perchloric acid)
 - ◆ Can be scaled up easily
- Excellent burr-removal
- Ra = 300-600 nm
- Pitting:
 - ◆ width < 6.5 μm
 - ◆ depth < 4.5 μm
- Gap increase $\sim 0.7 \mu\text{m/s}$ (10 μm / 15 s)



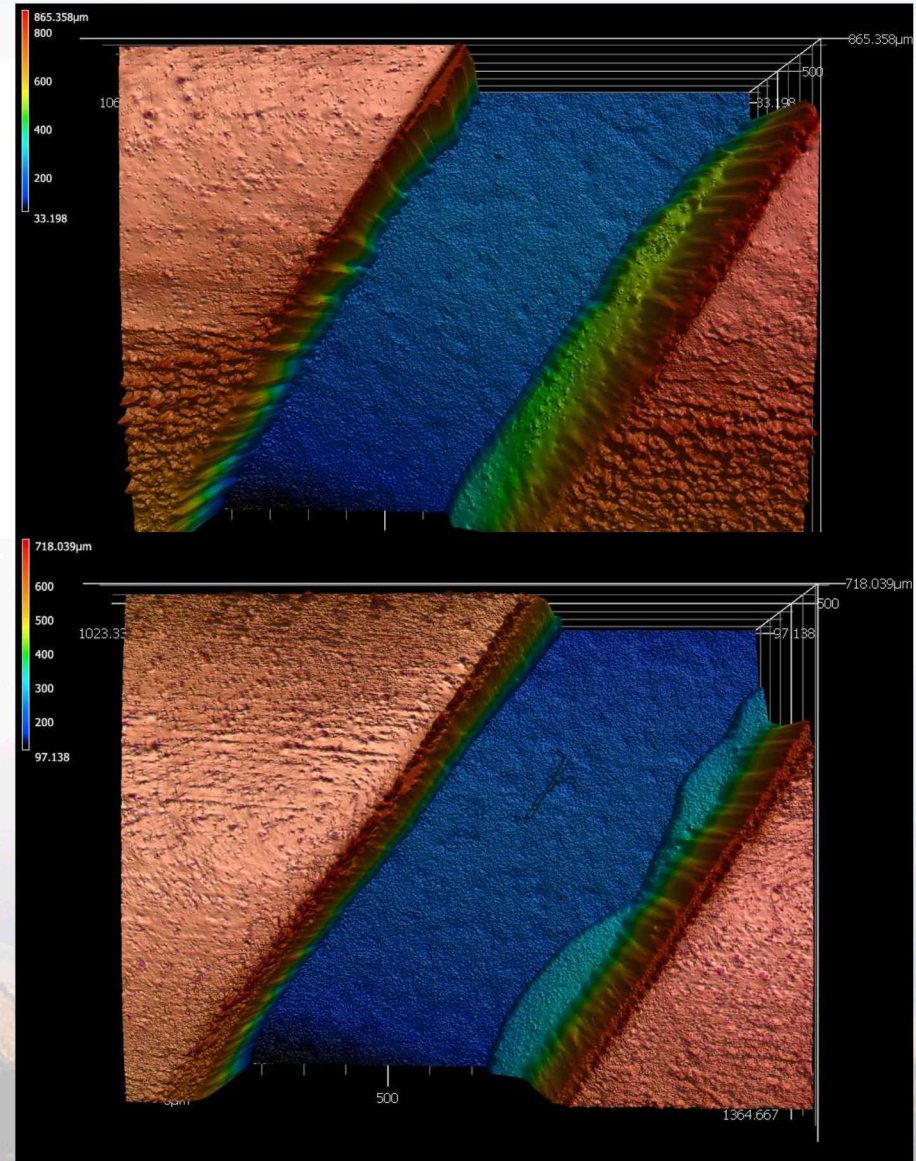
Non-Optimal Polishing Conditions

■ 50V, 11.55A, 3s on / 3s off, 350rpm agitation, distant electrodes, 40-60C, 150s

- Pulsing prevents mass-trans. ltd. removal
- High agitation increases impedance causing temp spikes & uneven surface from agitation gradient
- High temp does not selectively de-burr

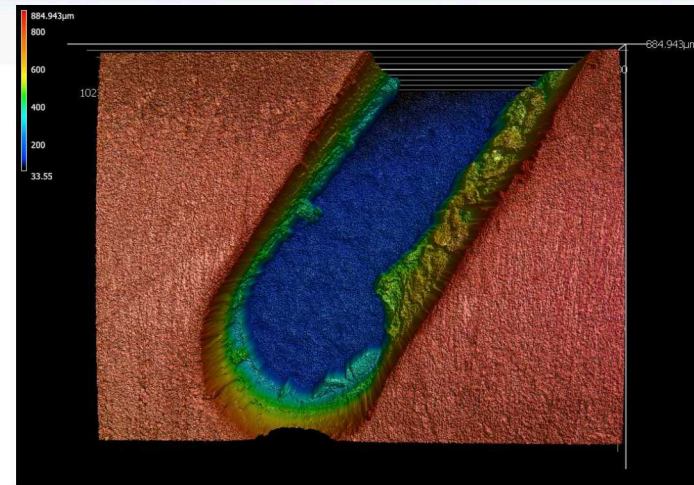
■ 65V, 15.75A DC, 25C, 120s

- High voltage is inefficient with high off-gassing
- High current increases temp fluctuation & removes material too quickly

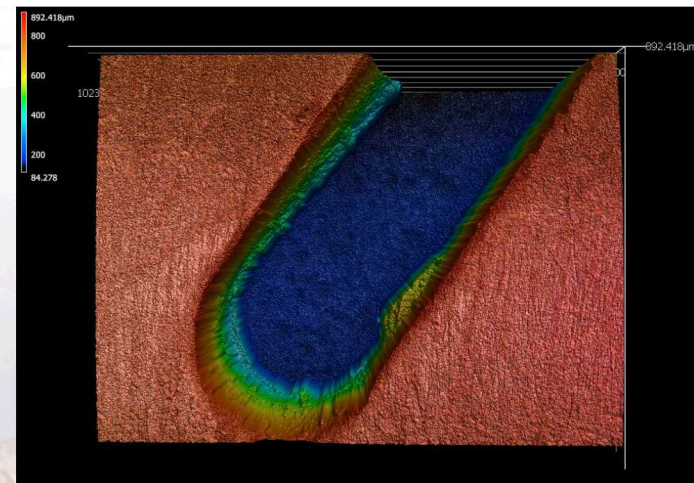


Summary of Process Improvements

- **Gap rough-pass with razor**
 - Clears excess material that is too large to be selectively removed from electropolishing
- **Ti wire “clamp”**
 - Prevents unwanted oxidation into chemistry
- **Curved counter electrode**
 - Radially symmetric polishing
- **Water bath**
 - Heat sink for stabilizing beaker temp
- **Lower agitation**
 - Uniform agitation, less impedance, & stabilizes temp
- **Low voltage**
 - Efficient & emits less heat



Prepolish



Razored Prepolish

Optimal Electropolishing of 500micron Targets

- **30 - 35 V**
- **11.55 A limit**
 - DC for 15 – 60 s depending on amount of material to be cleared
- **25 - 40 C**
 - Best results when CrO_3 is saturated and undissolved
- **150 rpm agitation**
 - Target submerged in chemistry directly above stir-bar (ensure adequate stirring between runs)
- **3-5 cm between target and counter electrodes**



Prepolish

15s

30s

40s

