MAB Phase Ceramics for Lunar Applications

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Advisor: Dr. Surojit Gupta of UND

2021 Summer Student Research Symposium

All images are courtesy of NASA/UND/Daniel Trieff unless otherwise stated

Outline

- ❖About Me
- ❖Objective
- ❖Background on SLM
- ❖Approach
- ❖Results
- ❖Analysis/Summary
- ❖Next Steps
- ❖Acknowledgements

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About Me

- ❖Senior at the University of North Dakota studying Mechanical Engineering
- ❖Past work experience includes: Ulliman Schutte, R3 Aerospace, Cirrus Aircraft, and researcher at UND
- ❖I'm a pilot and an avid scuba diver

Images courtesy Daniel Trieff

Objective

- ❖Overall objective: manufacture highly wear resistant and corrosion resistant material for lunar applications
- ❖Design MAB powders with tailored flowability (Done by Dr. Gupta and Mackenzie Short with my assistance)
- ❖Synthesize 3D printed MAB structures using SLM (Selective Laser Melting) process (Done by Dr. Hocker)

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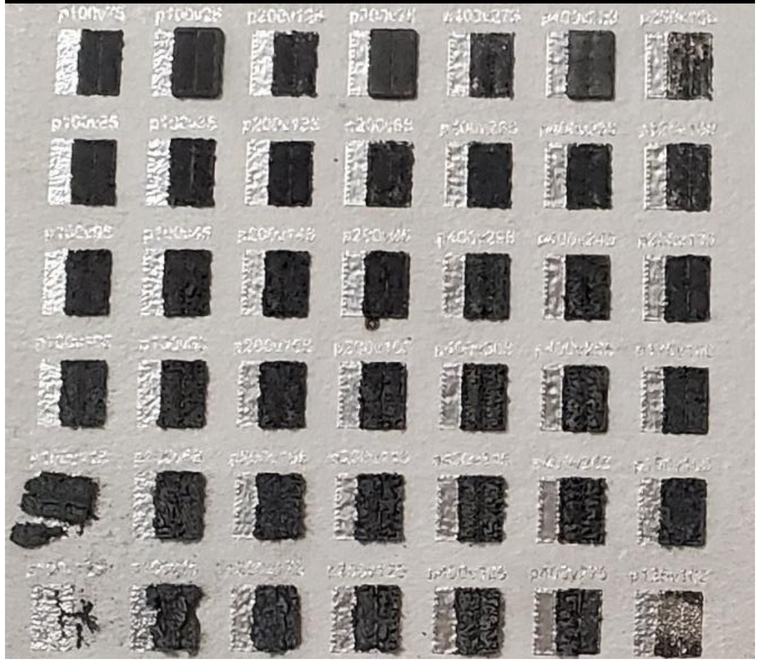
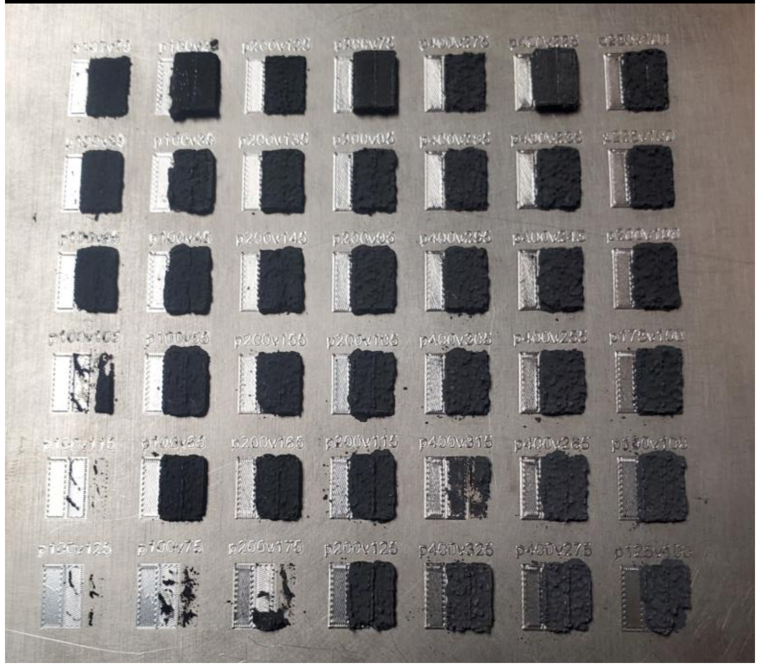
Objective

❖Design a characterization paradigm for understanding the feasibility of MAB phase films for wear resistant applications (Done by Daniel Trieff and Dr. Gupta with assistance from Mackenzie Short)

MAB 3D Printed on Steel Plate



MAB 3D Printed on Steel Plate



Background on Selective Laser Melting

- ❖Selective Laser Melting is an additive manufacturing technique by which a powder bed is melted and forms a solid structure [1]
- ❖Another layer of powder is added and the process repeats
- ❖We are using SLM to 3D print MAB phase powders for lunar applications [1]
- ❖The variables NASA manipulated were wattage and scanning speed

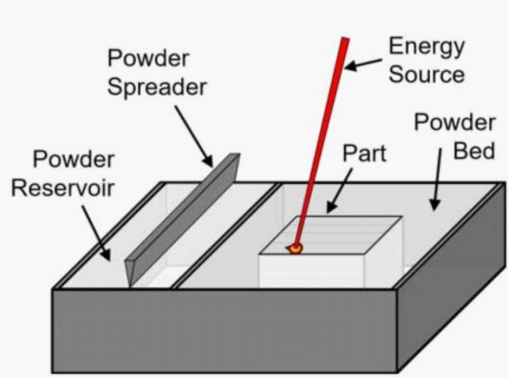


Image courtesy of Dr. Brodan Richter

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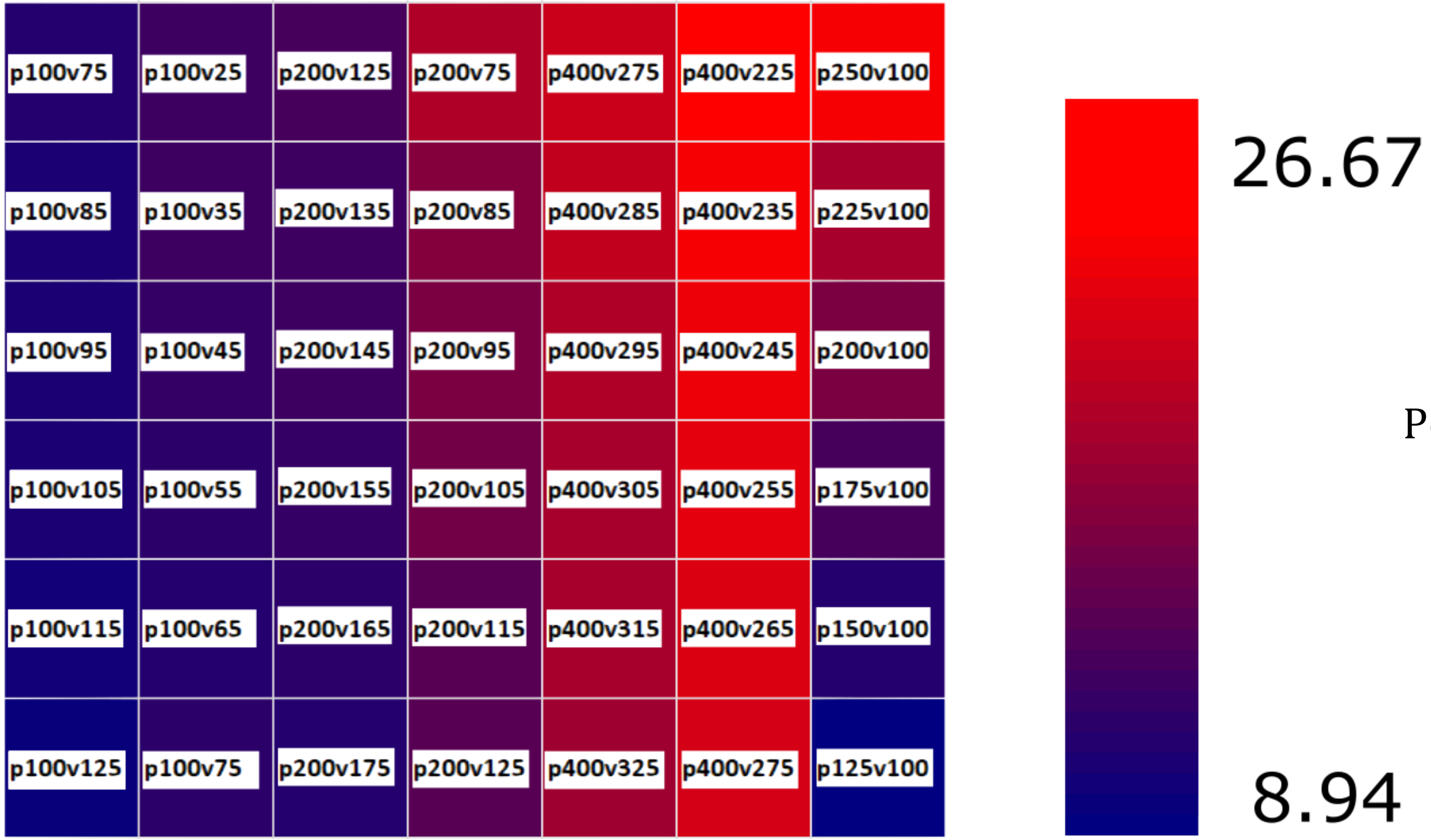
Approach (Characterization Paradigm)

- Visual Inspection
  - Quickly able to rule out the pieces that had broken off the plate
- Optical Inspection
  - Observed samples as received under microscope
- Profilometry
  - Used stylus profilometer to gather data on surface roughness
- Sonication
  - Used ultrasonic bath to clean samples
- Optical Inspection Post Cleaning
  - Observe samples after being cleaned and polished to observe microstructure
- SEM (Scanning Electron Microscope)
  - Use backscattered and secondary imaging to see if there is decomposition present

Power Densities



$$\text{Power density} = \frac{power(W)}{\sqrt{velocity(mm/s)}}$$

Color mapping from modified equation from King et al. [2]

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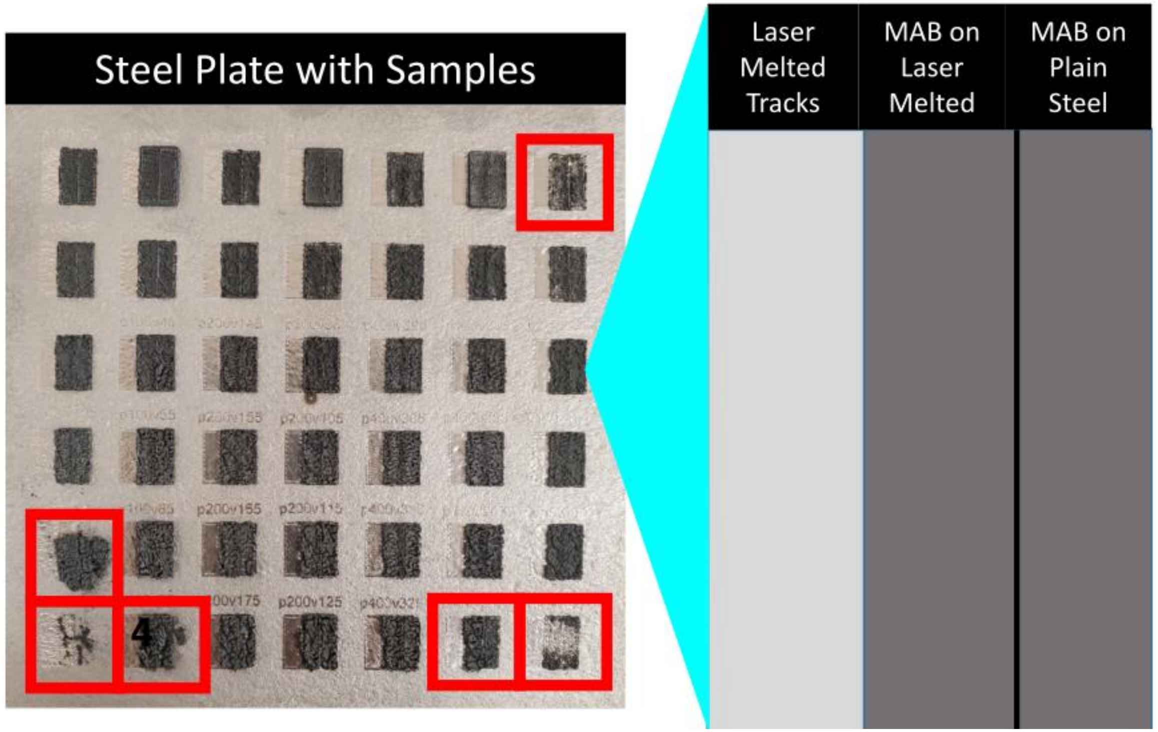
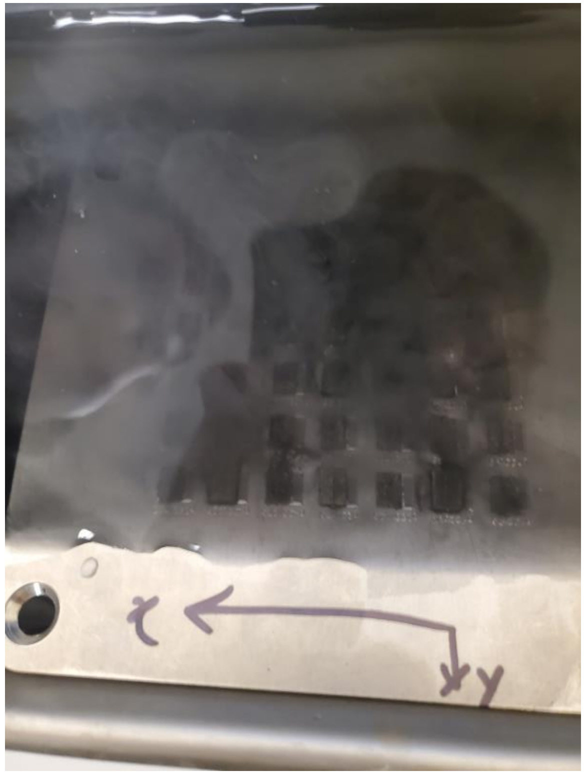
Screening of 3D printed MAB phase by Visual Inspection



❖We took note of which samples were broken off and ruled them out

Cleaning the Samples

❖Branson 2800 Series Ultrasonic Cleaner
❖Ran for 5 minutes in distilled water (first attempt was for 60 minutes)



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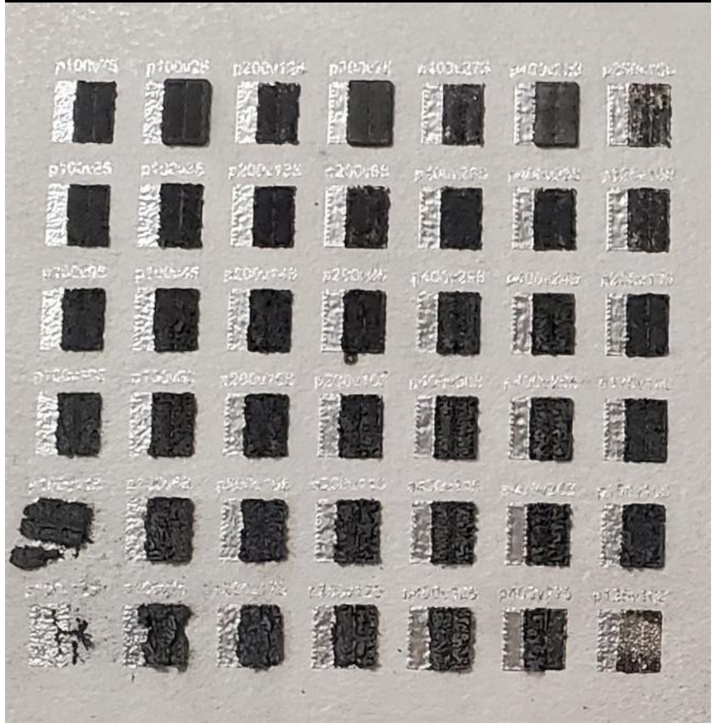
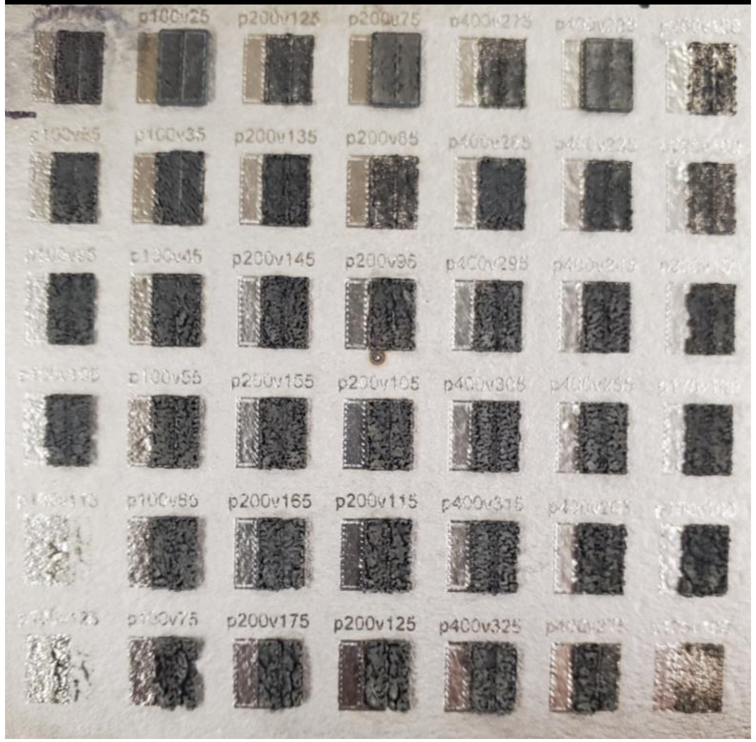
Optical Inspection Post-Cleaning

- We then sonicated the plates to see the microstructure without the MAB debris/dust layer on the surface

MAB on Steel Plate Pre-Cleaning



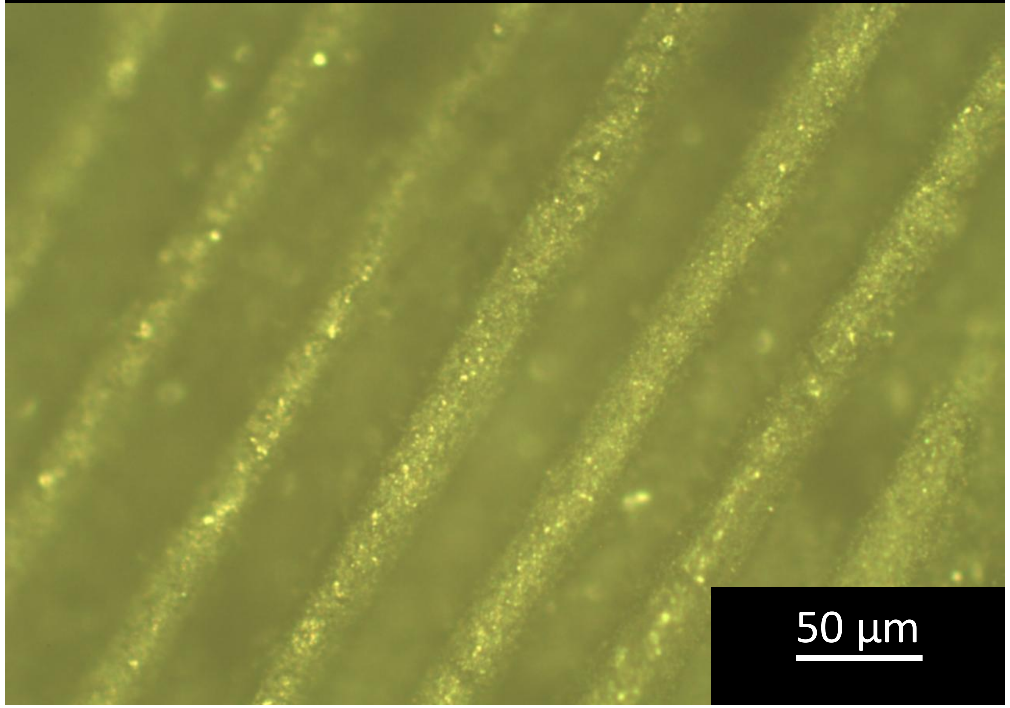
MAB on Steel Plate Post-Cleaning



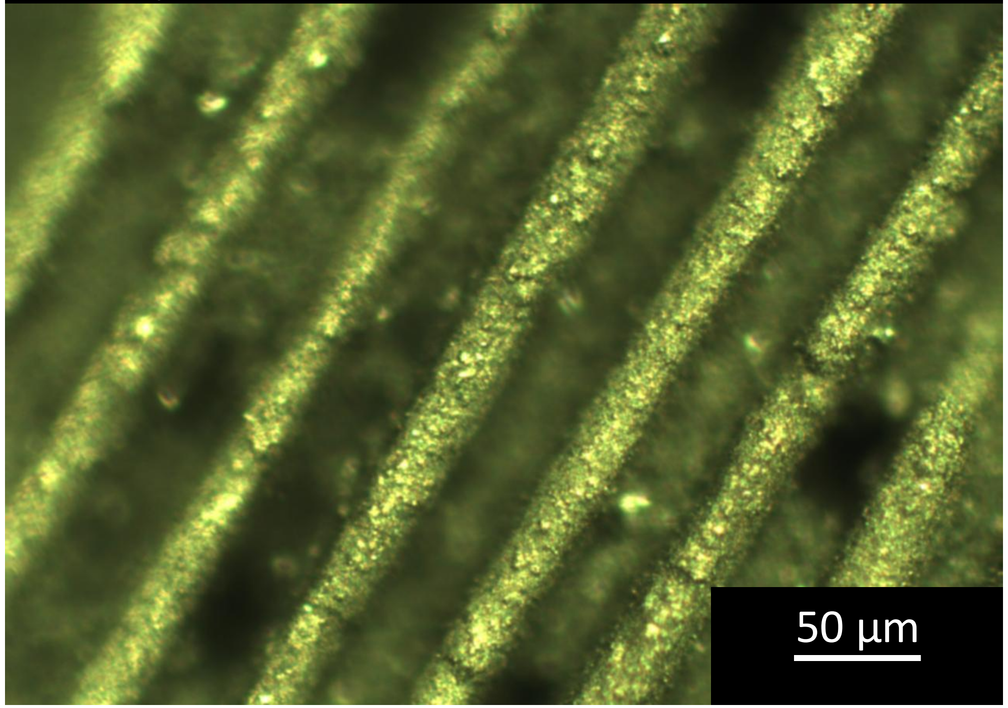
Optical Inspection

- We then did optical microscopy of the MAB phases on the plates
  - This gave a preliminary idea of porosity and other surface features

Layered on Plain Steel Surface Bright Field



Layered on Plain Steel Surface Dark Field



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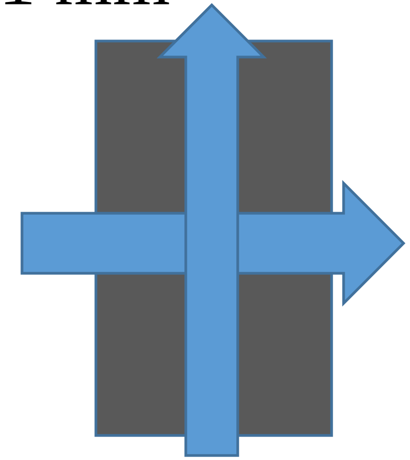
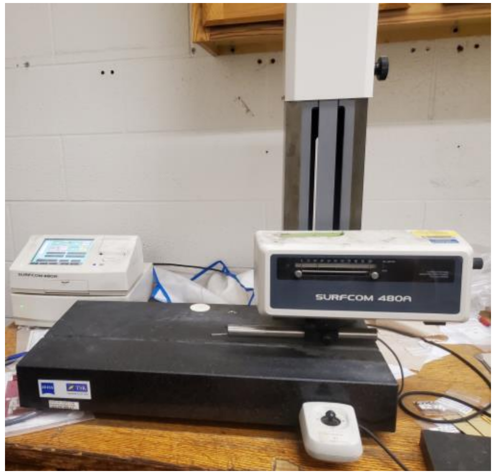
Profilometry

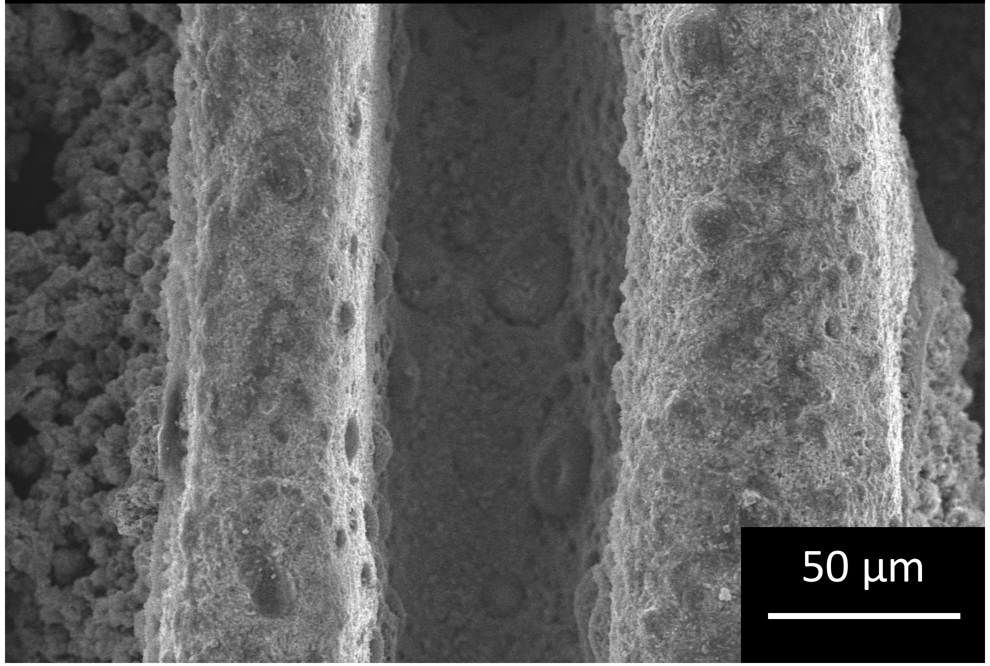
- We wanted the samples to be less than a given surface roughness
  - Smoother samples would correlate to lower porosity which leads to higher density
- To determine the surface roughness, a stylus profilometer was used
- We ran the stylus across the middle of each sample in two directions
- The evaluation length was 1 mm



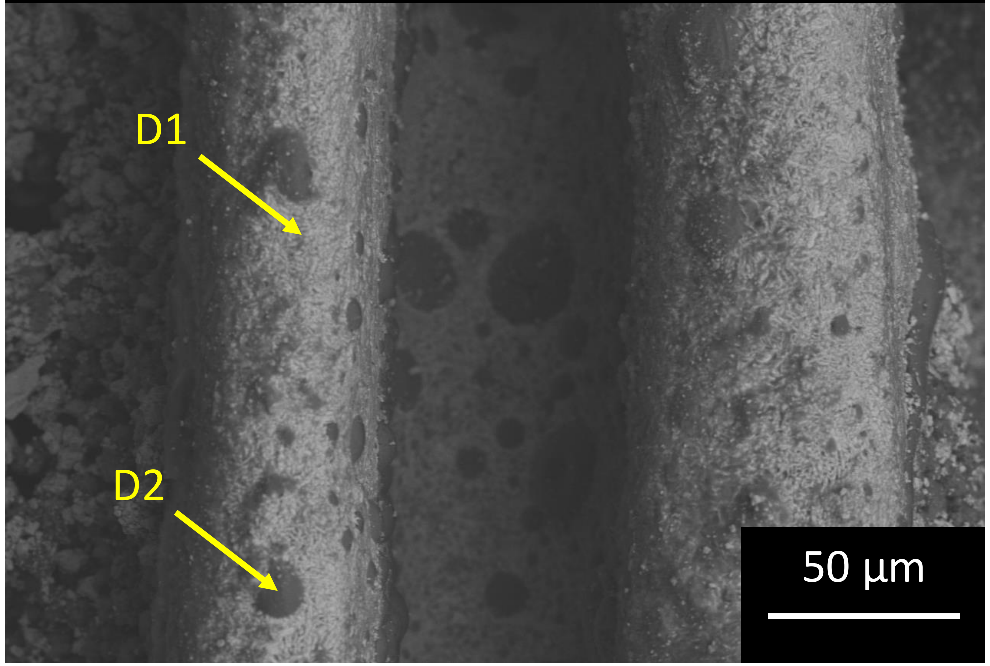


Backscattered Electron SEM (Scanning Electron Microscope)

Secondary Electron

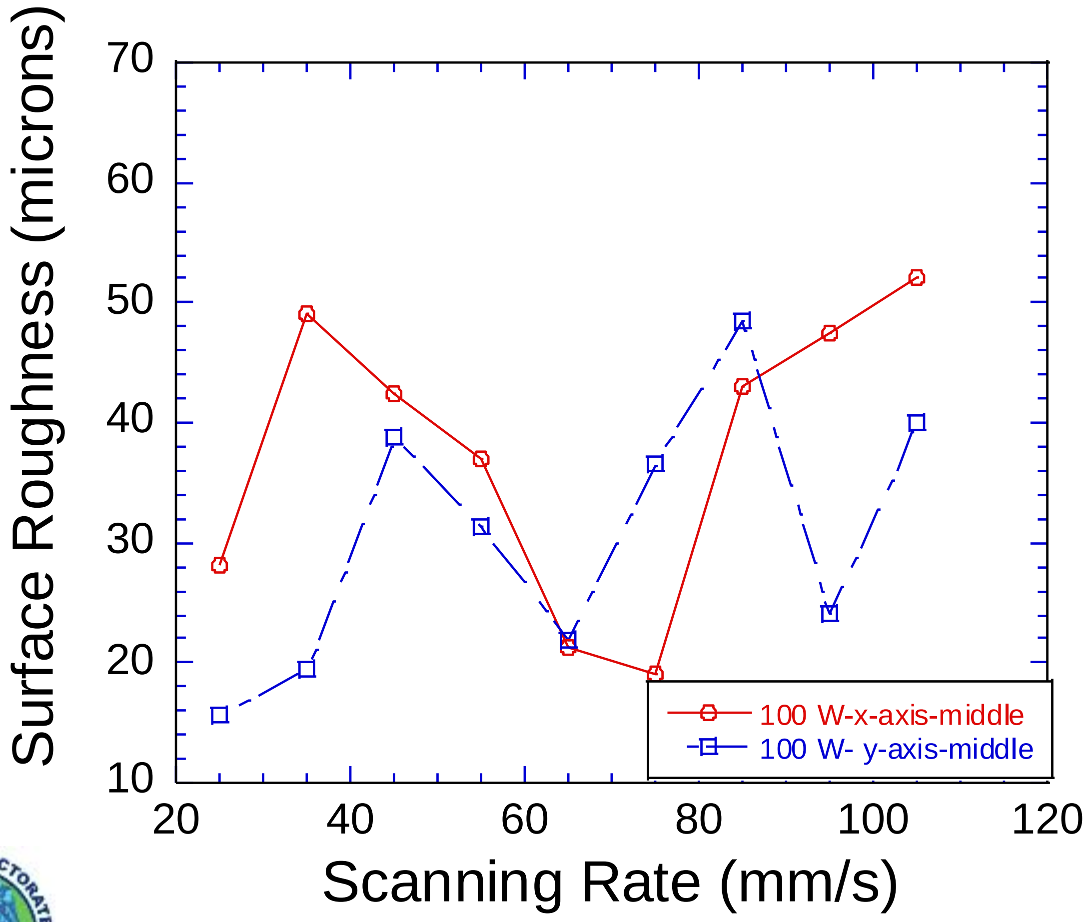


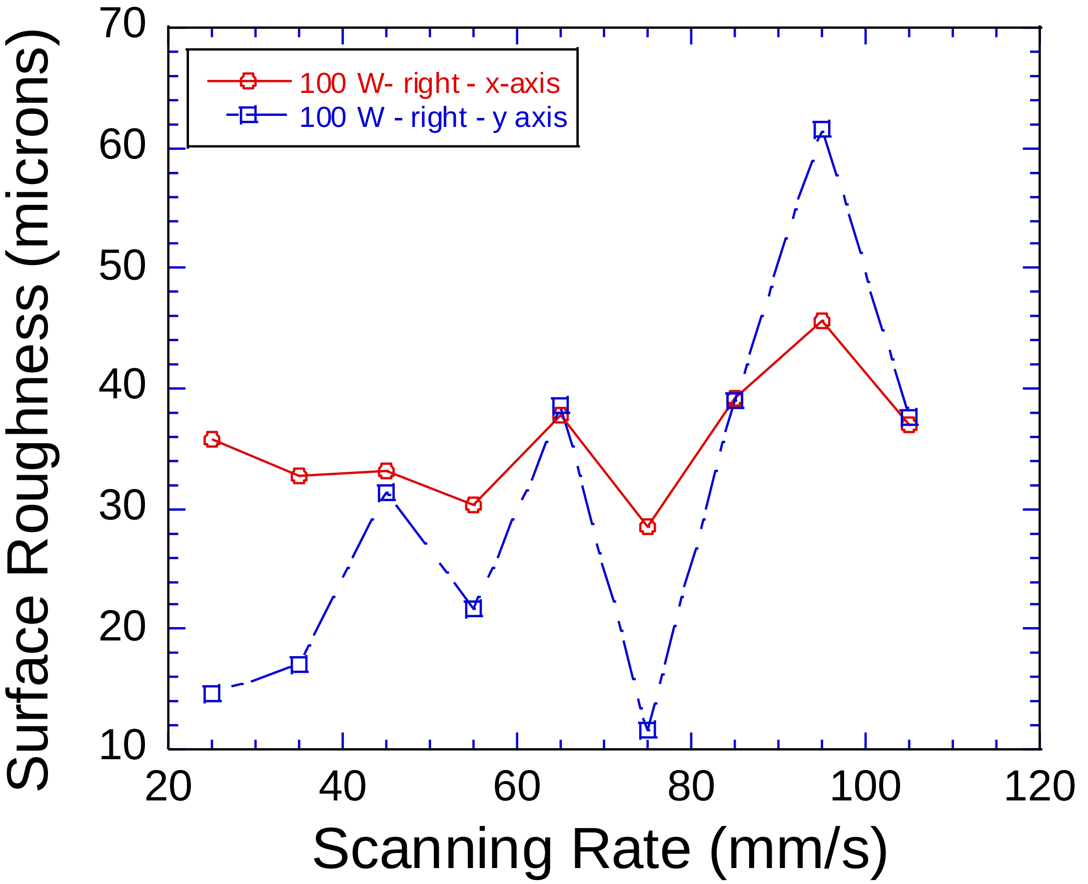
Backscattered Electron



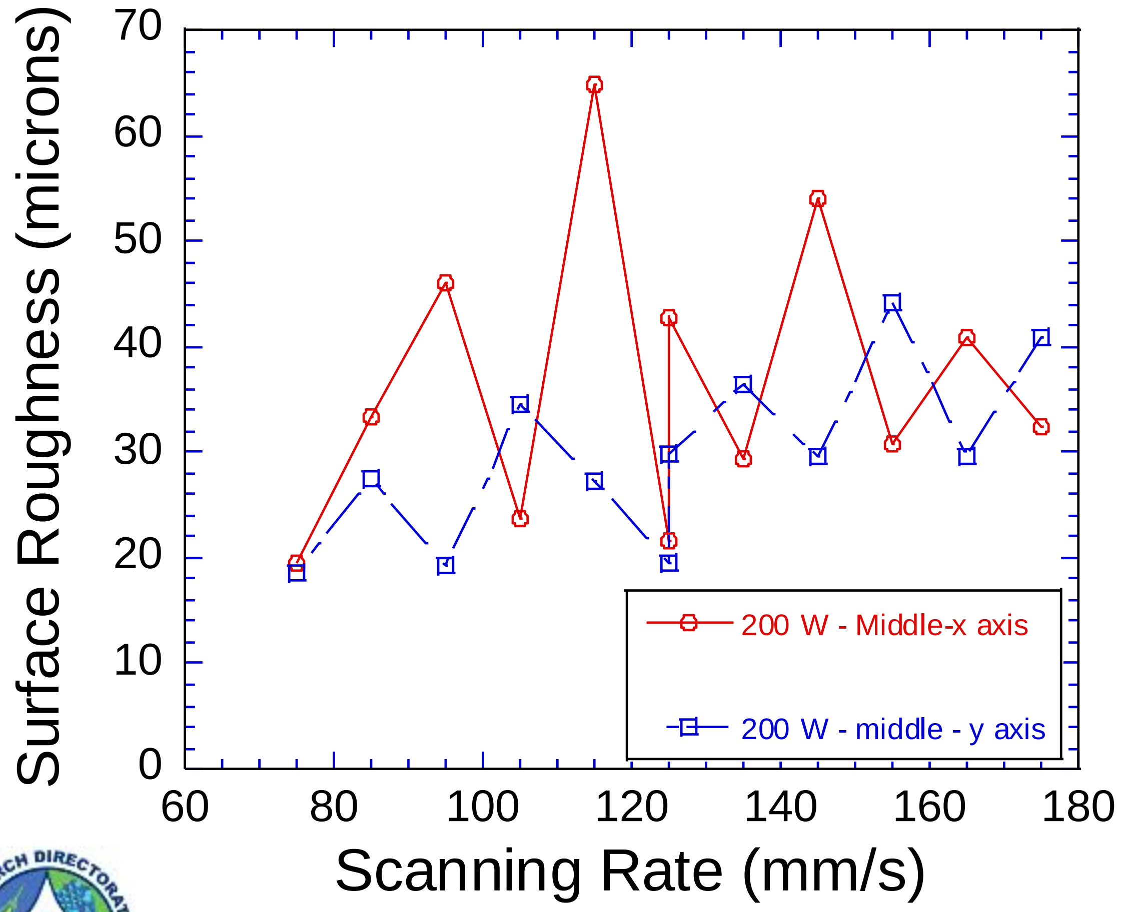
Images from Daniel Trieff/Dr. Gupta

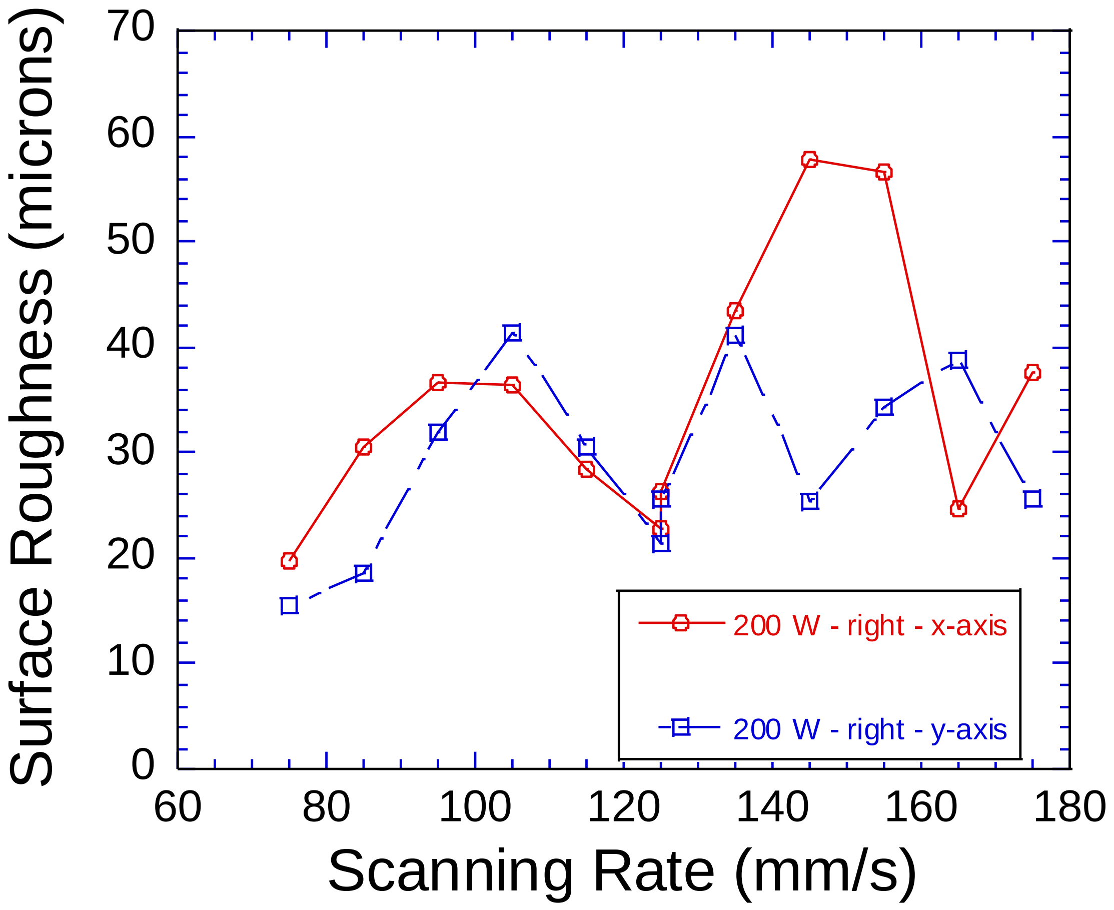
Profilometry Results: MAB on Steel Plate 100 Watts Pre-Cleaning



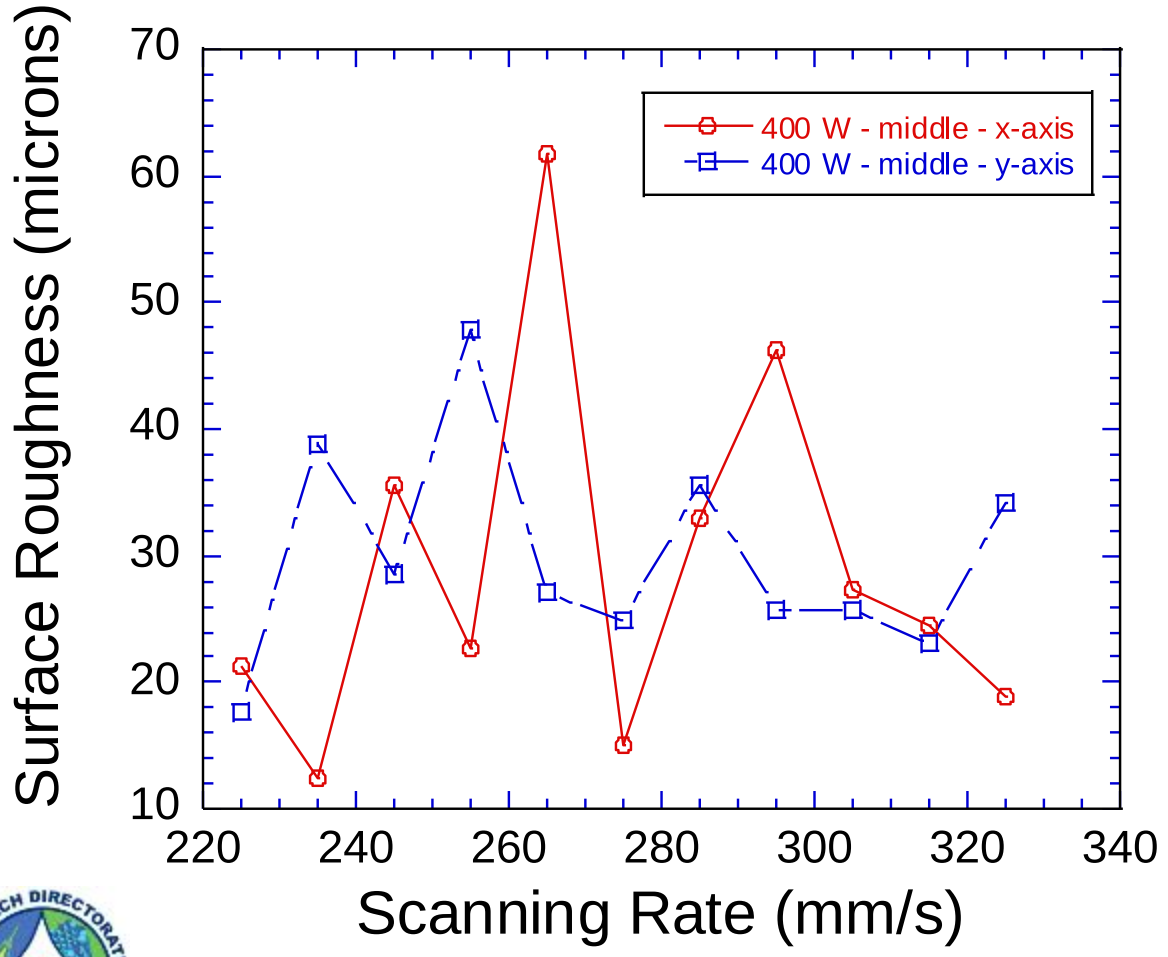


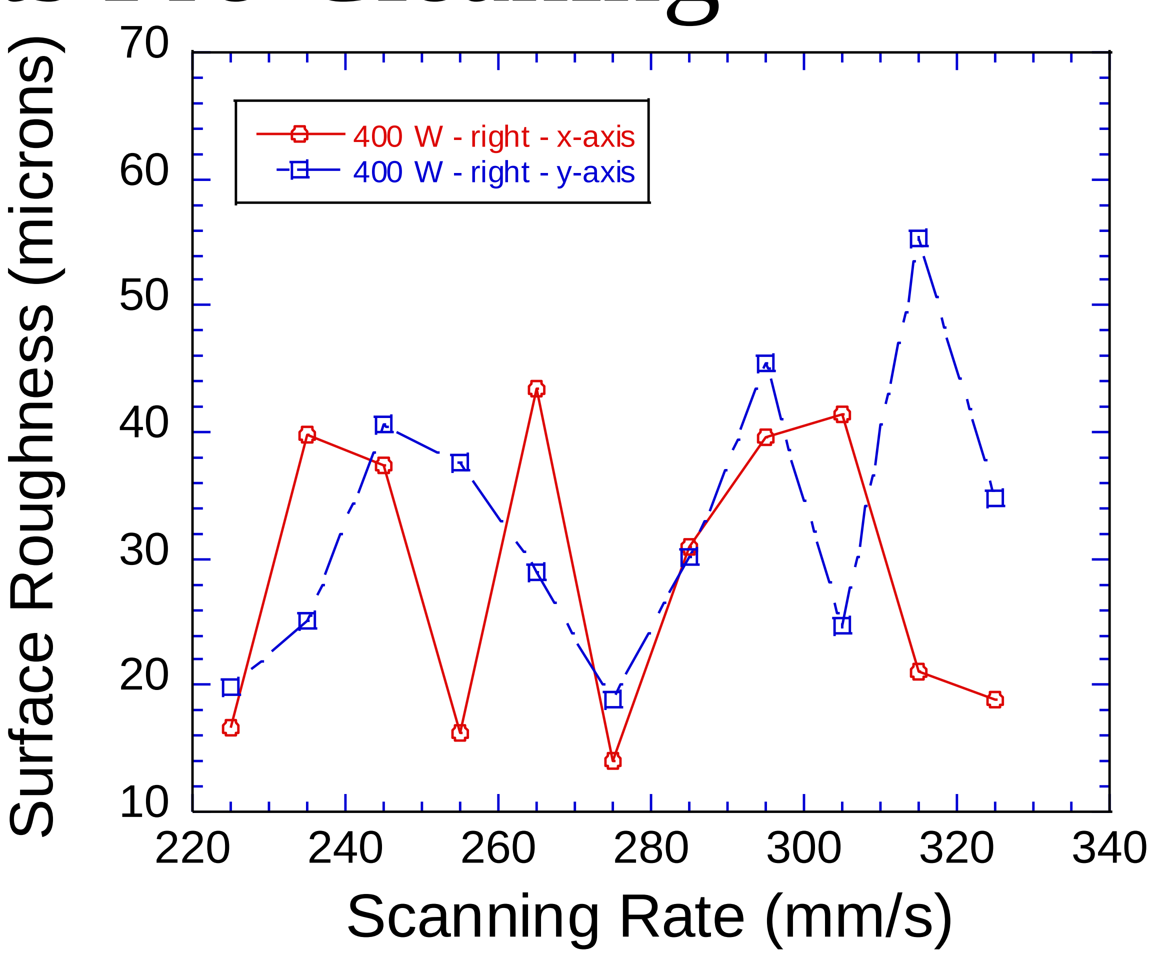
Profilometry Results: MAB on Steel Plate 200 Watts Pre-Cleaning





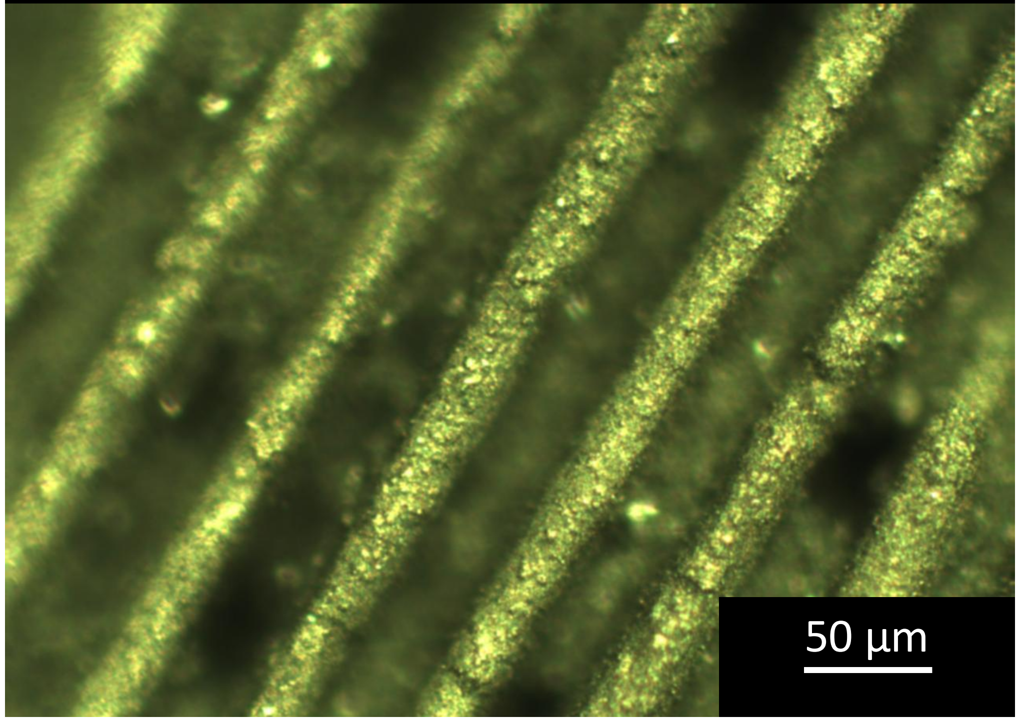
Profilometry Results: MAB on Steel Plate 400 Watts Pre-Cleaning



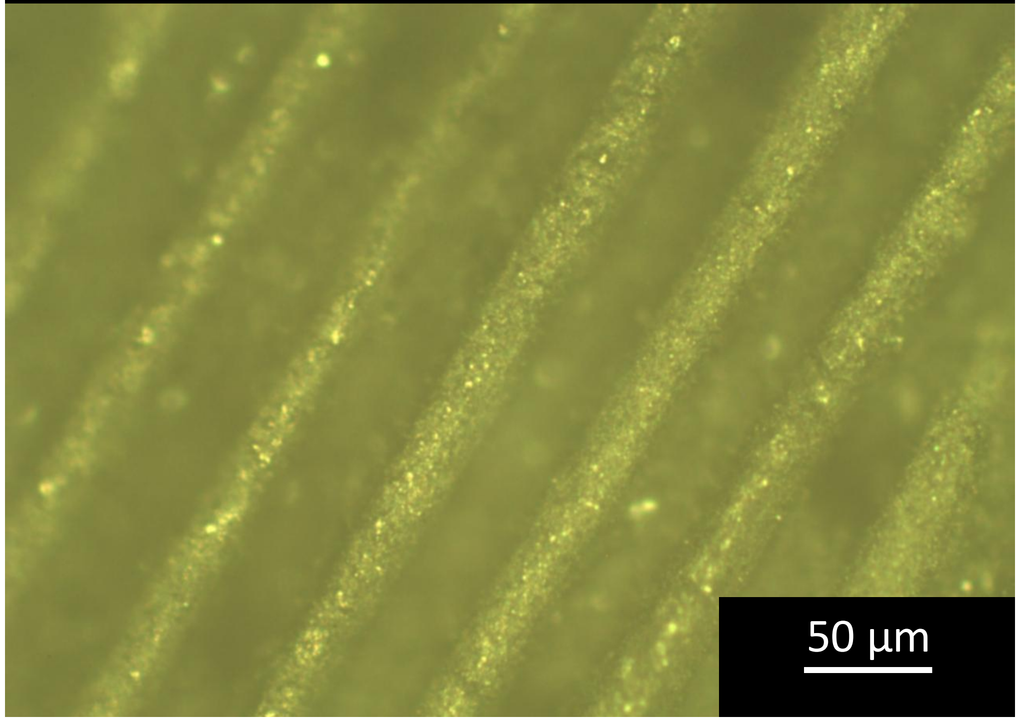


Optical Microscopy: MAB on Steel p100v25R at 10x

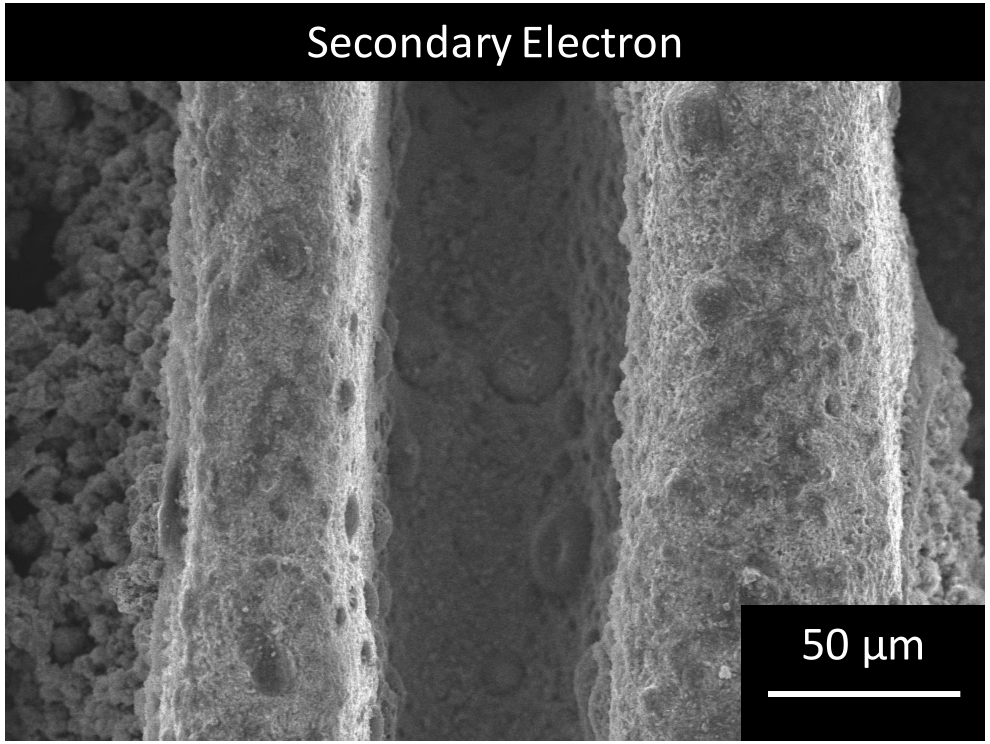
Layered on plain steel surface with Dark Field

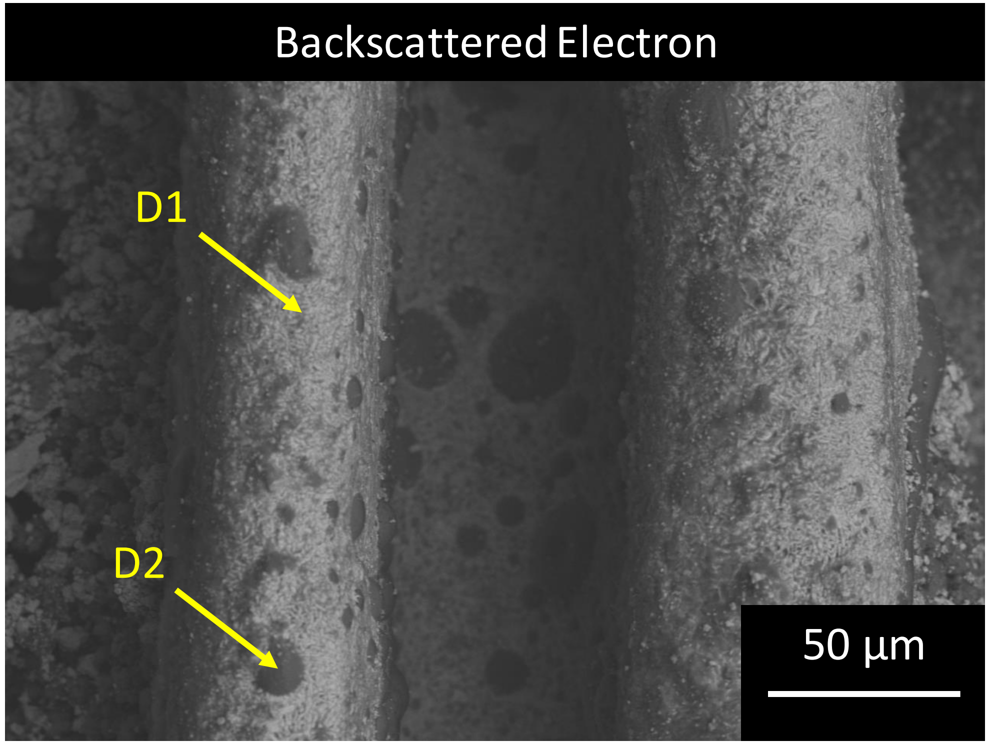


Layered on plain steel surface with Bright Field



SEM Results: MAB on Steel p100v25M



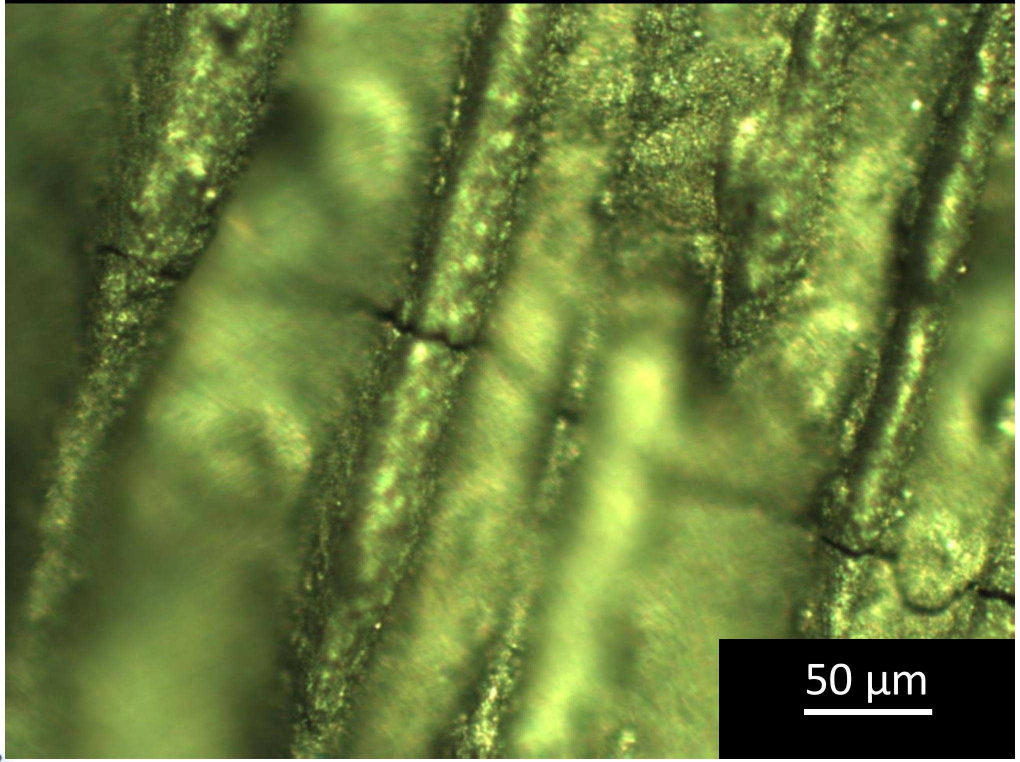


	Mo	Al	O	Mo/AL
D1	24.557±.587	24.147±.493	45.240±2.017	1.017
D2	0.980±.143	58.153±.770	36.277±1.033	0.017

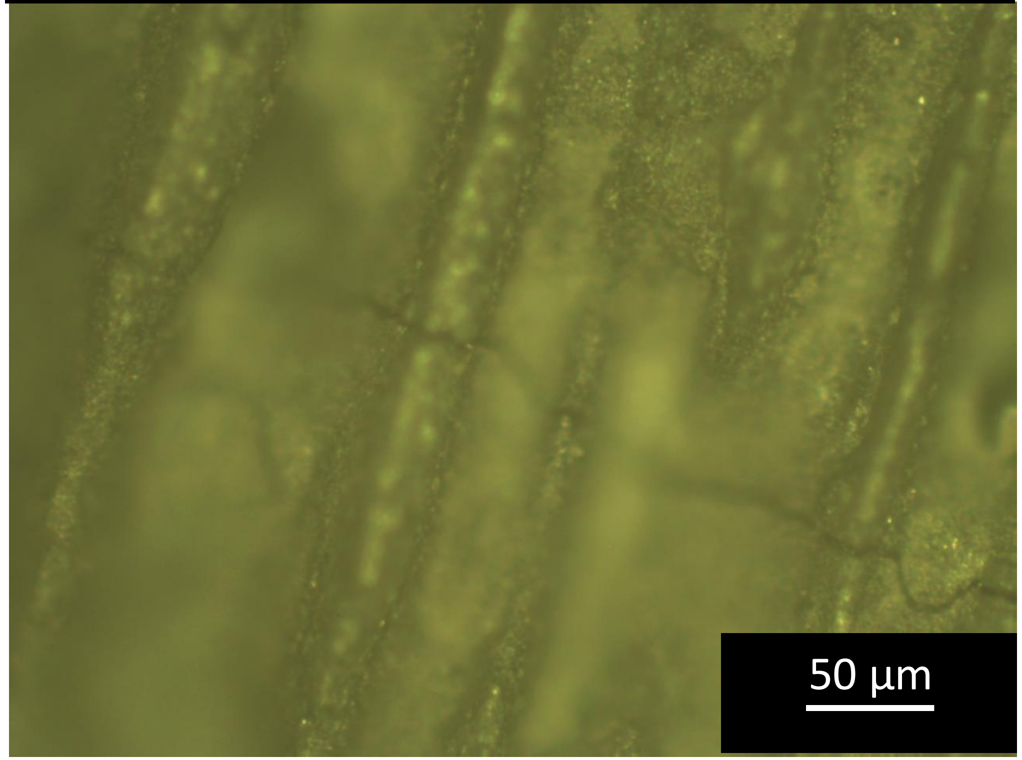
Images courtesy Daniel Trieff/Dr. Gupta

Optical Microscopy: MAB on Steel p200v75M at 10x

Layered on etched surface Dark Field



Layered on etched surface Bright Field



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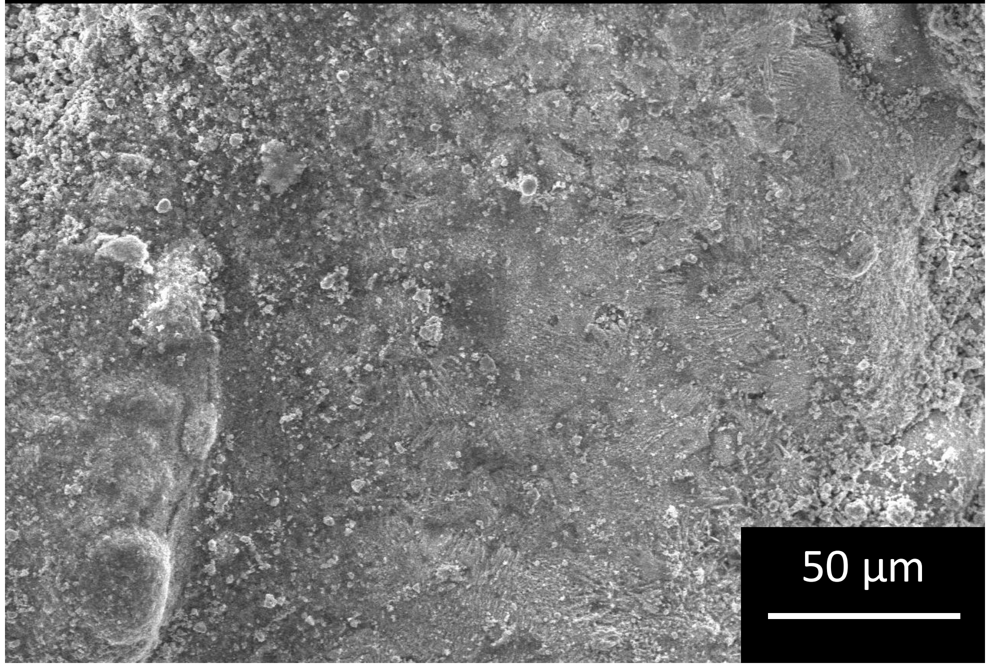
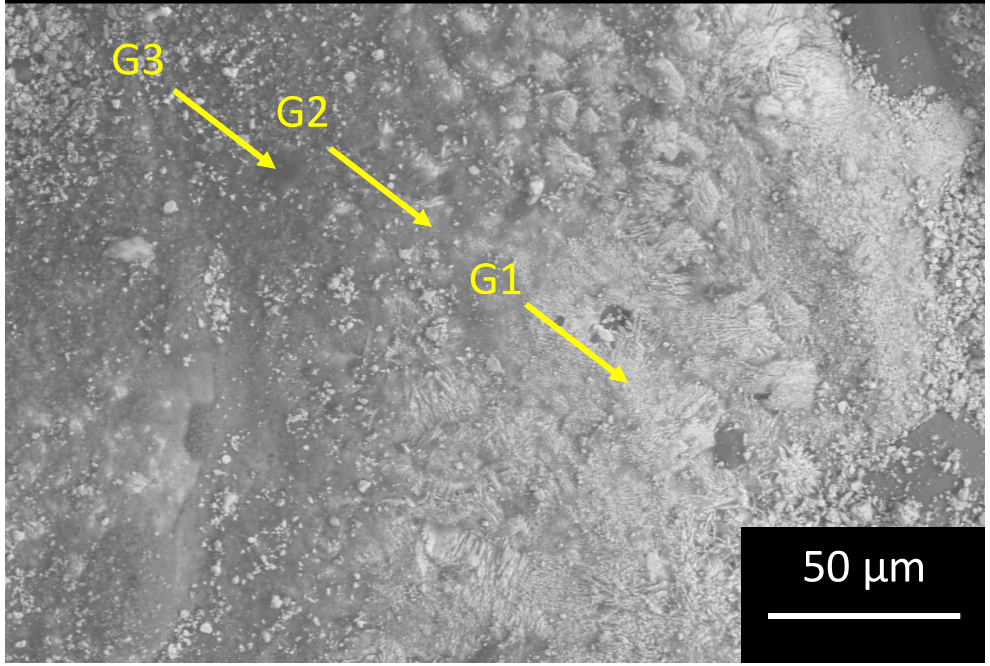
SEM Results: MAB on Steel p200v75M

Secondary Electron



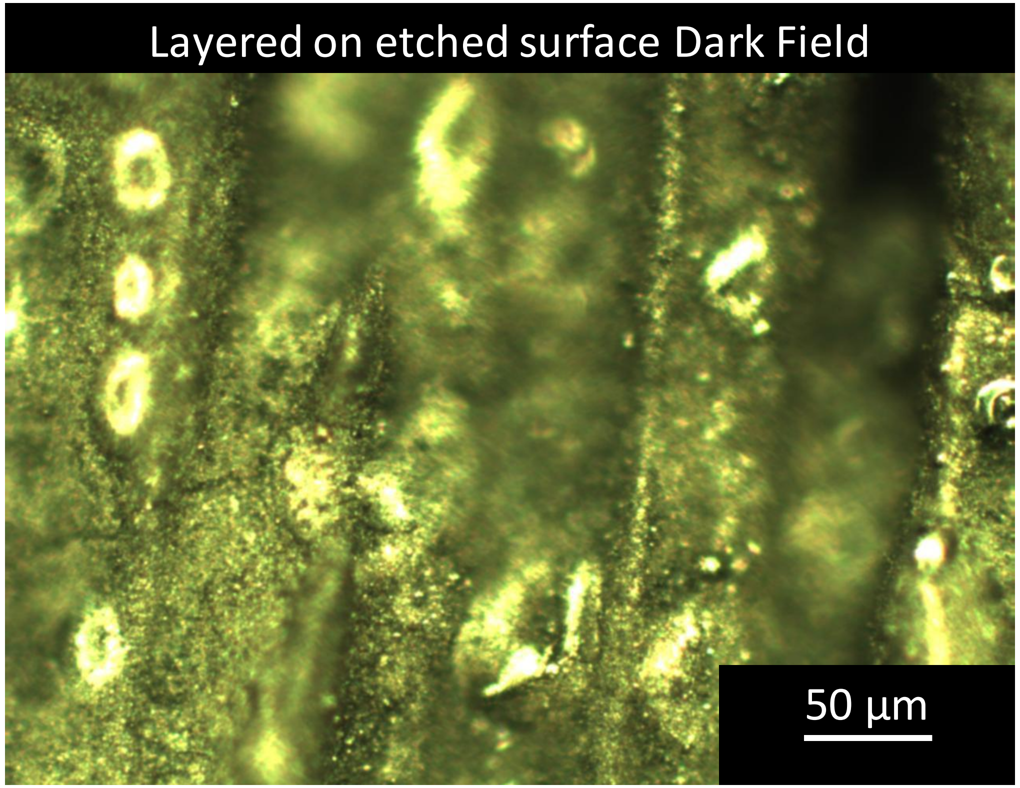
Backscattered Electron

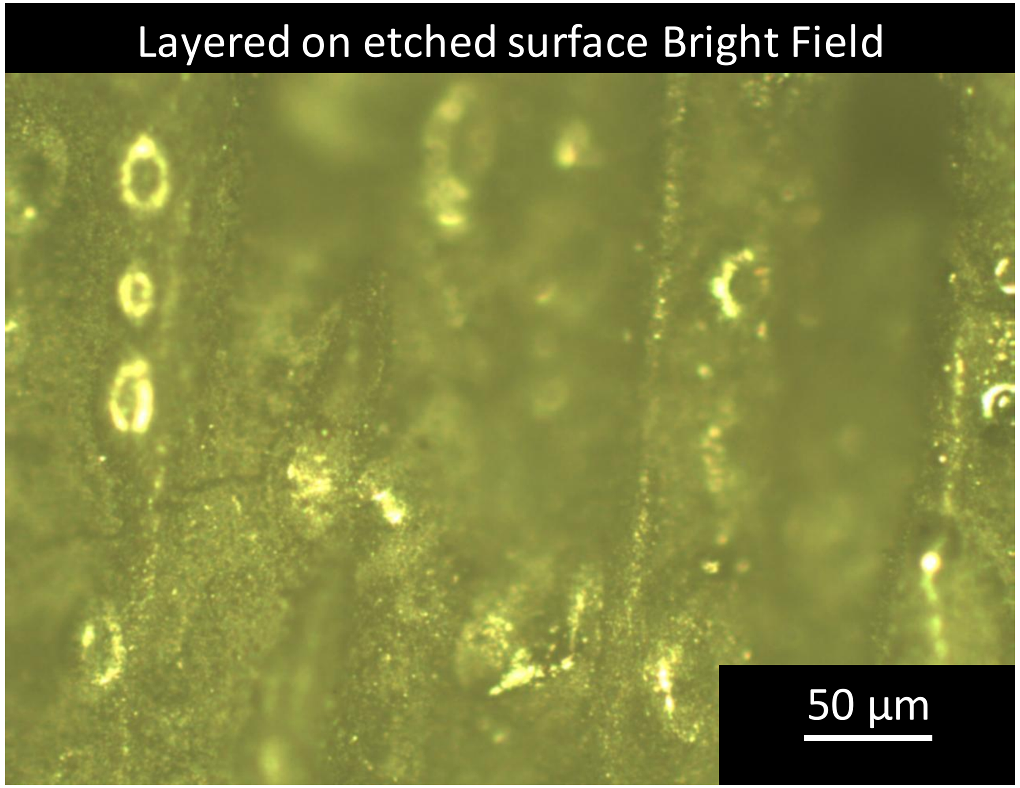


	Mo		Al		O		Mo/AL
G1	27.613	±.667	30.173	±.560	37.663	±1.580	0.915
G2	7.830	±.240	46.283	±.610	40.980	±1.240	0.169
G3	0.420	±.060	15.617	±.237	14.090	±1.070	0.027

Images courtesy Daniel Trieff/Dr. Gupta

Optical Microscopy: MAB on Steel p400v225M at 10x





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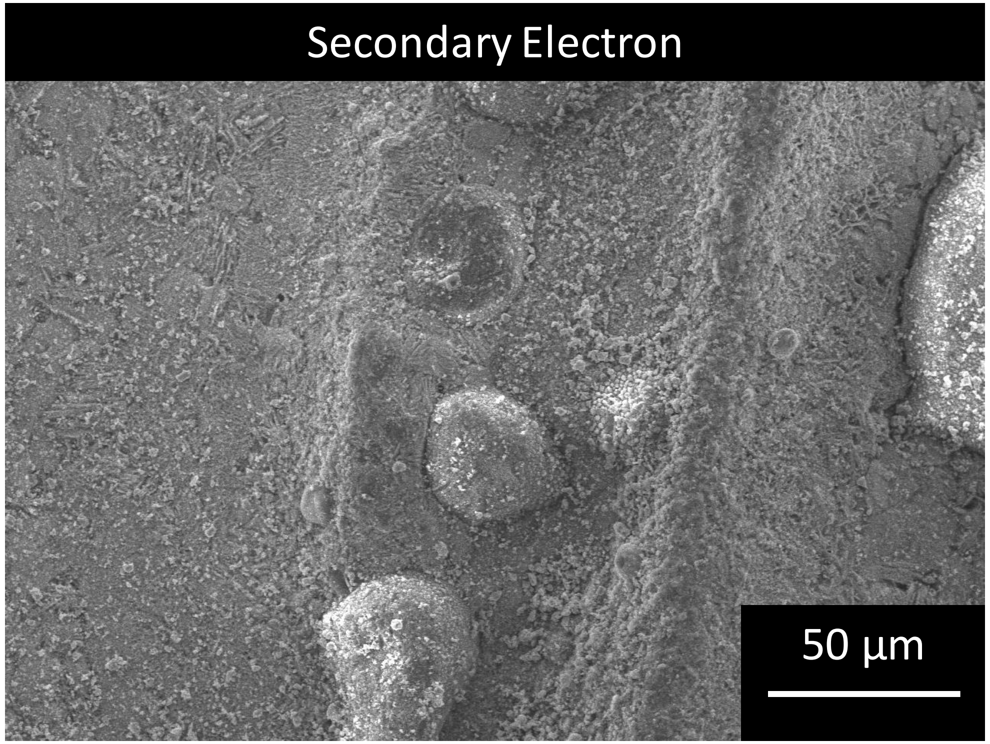
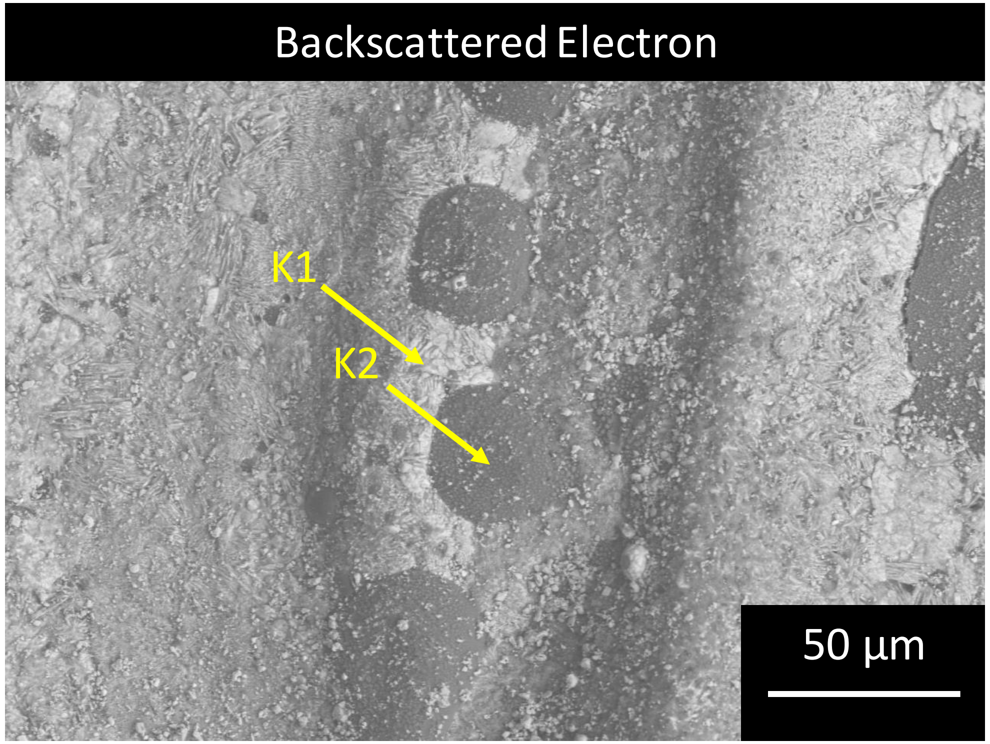
SEM Results: MAB on Steel 400v225M





	Mo		Al		O		Mo/AL
K1	29.360	0.610	25.233	±.497	40.907	±2.010	1.164
K2		±	48.627	0.607	50.333	±1.023	0.000

Images courtesy Daniel Trieff/Dr. Gupta

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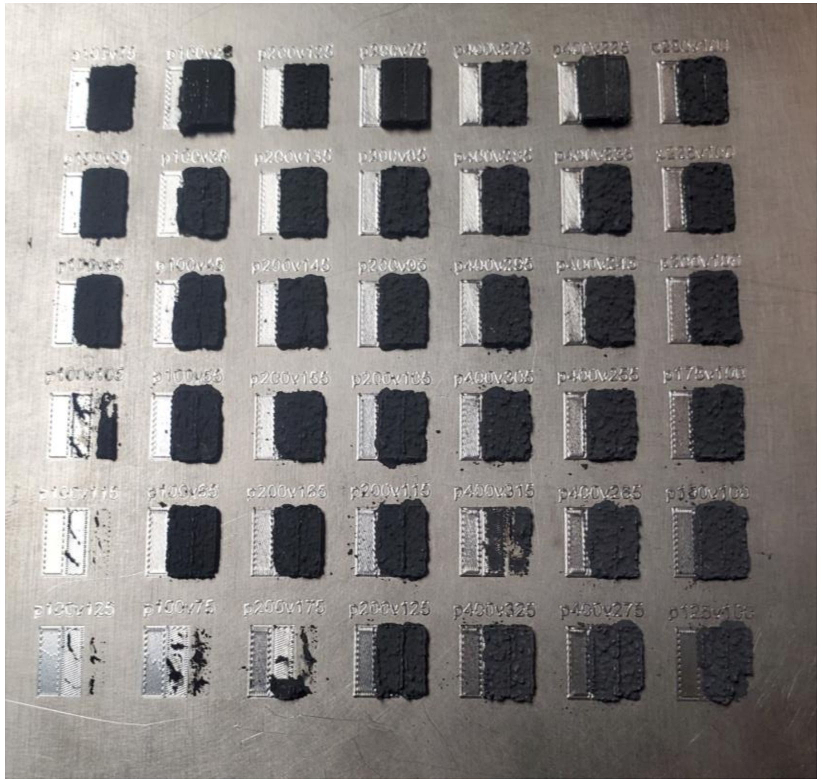
MAB Printed on Titanium Results

❖ Briefly discuss differences with Titanium procedure and results



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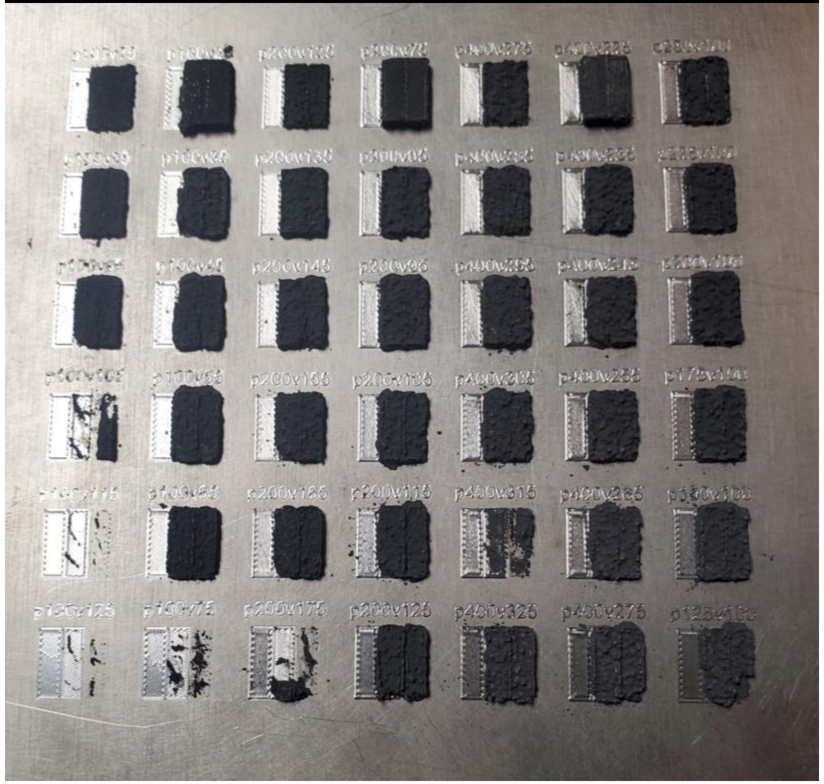
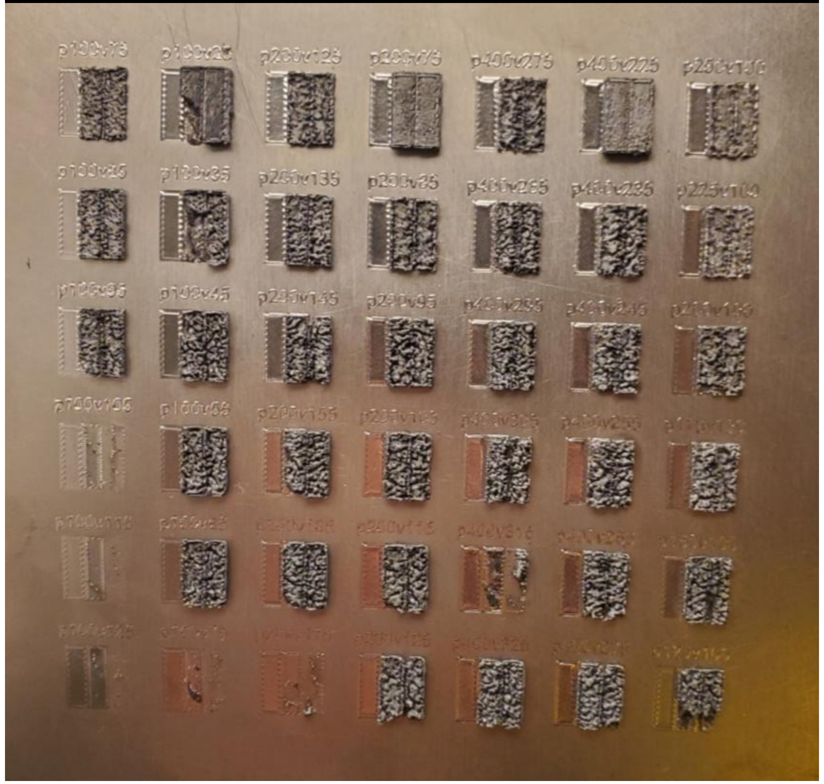
Results of Sonication at 60 Minutes

MAB on Titanium Plate Pre-Sonication

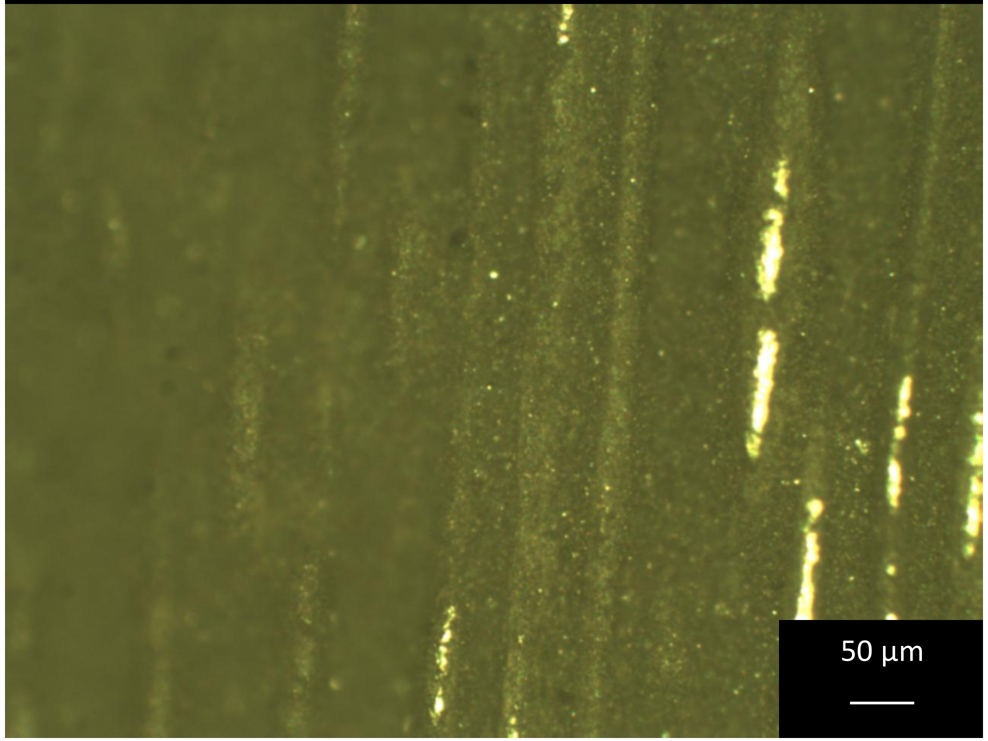


MAB on Titanium Plate Post-Sonication

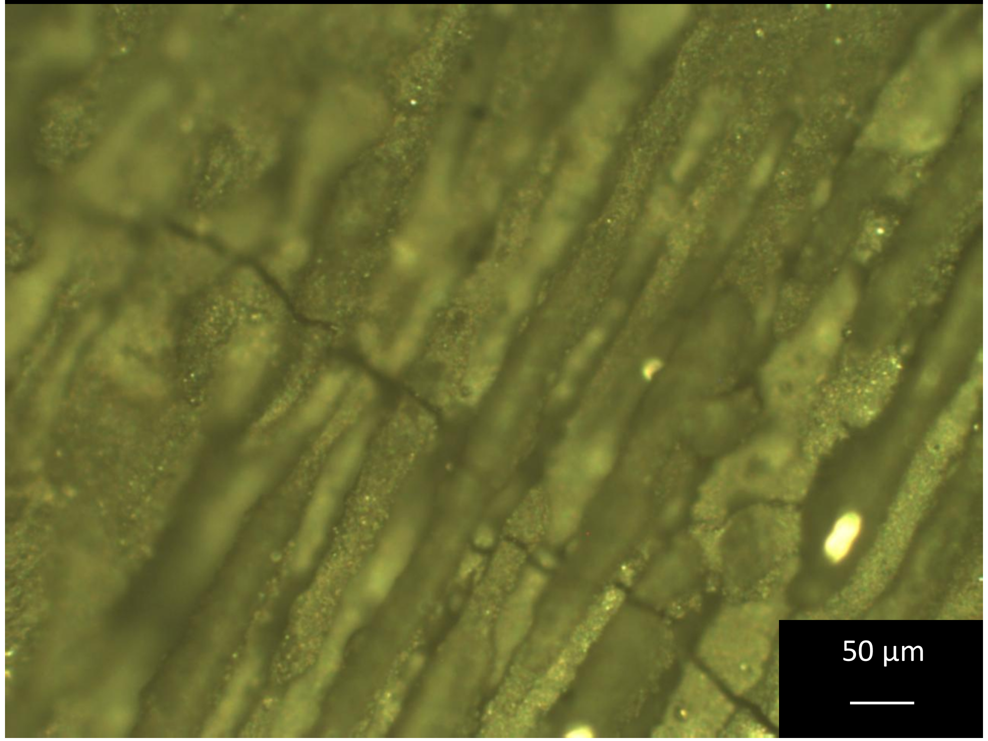


Results of Optical Microscopy

Ti-p100v25M Pre-Clean



Ti-p100v25R Post-Clean



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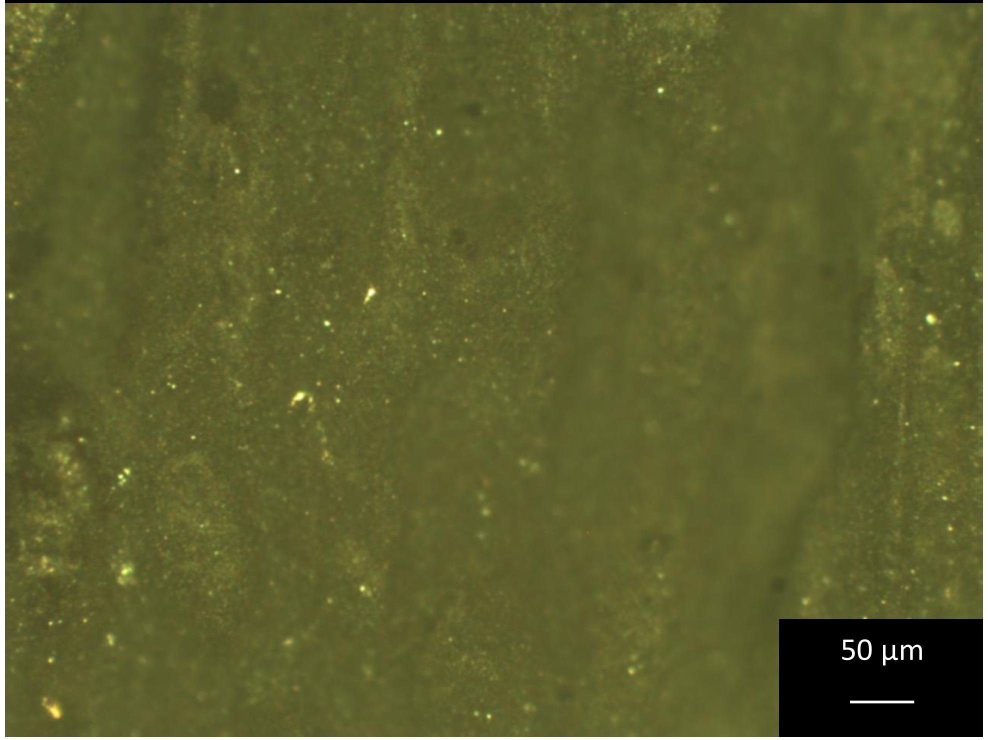
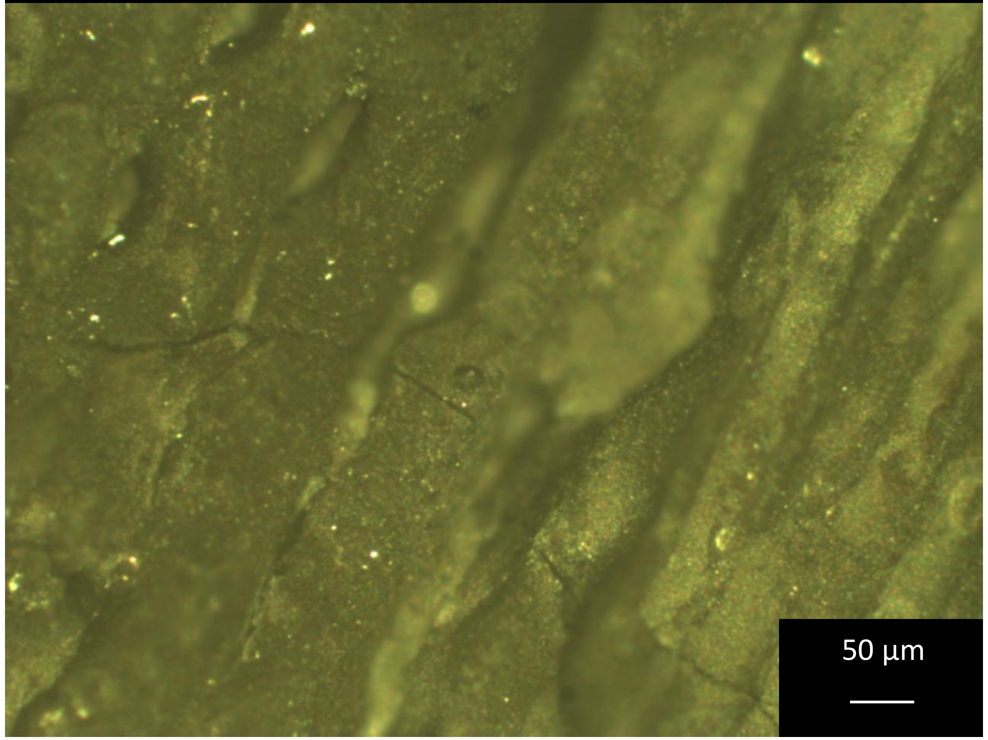
Results of Optical Microscopy

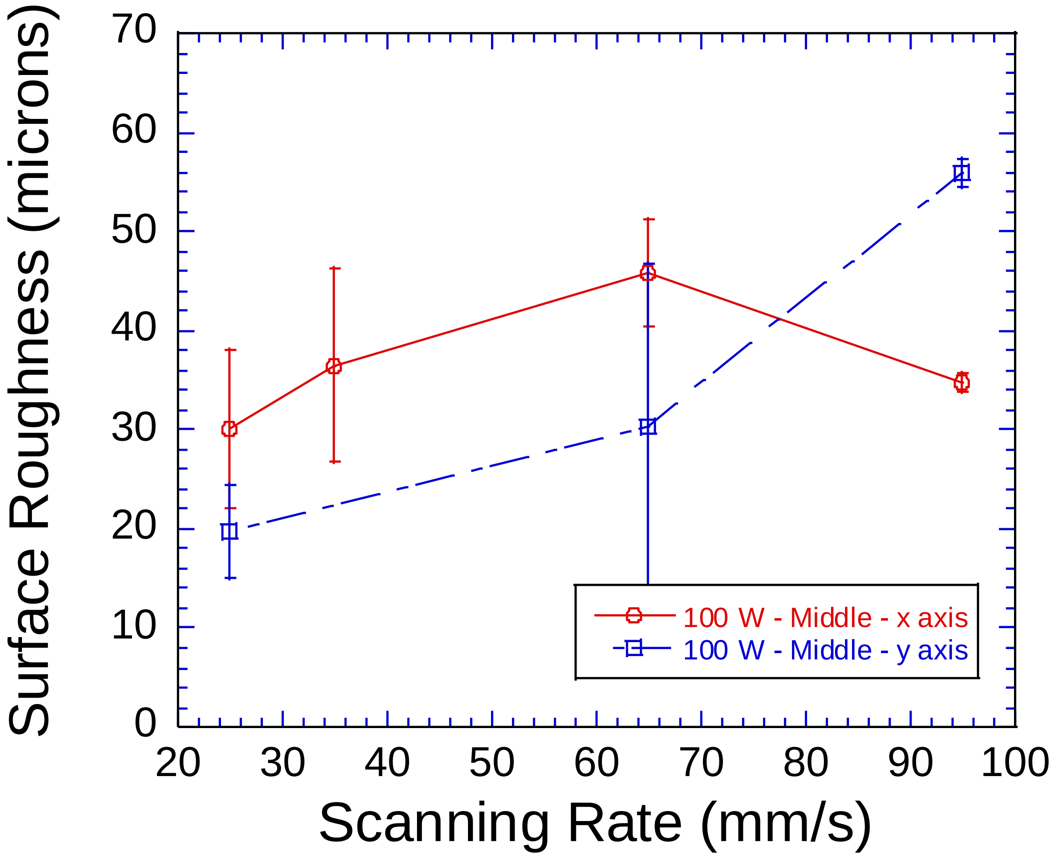
Ti-p200v75M Pre-Clean



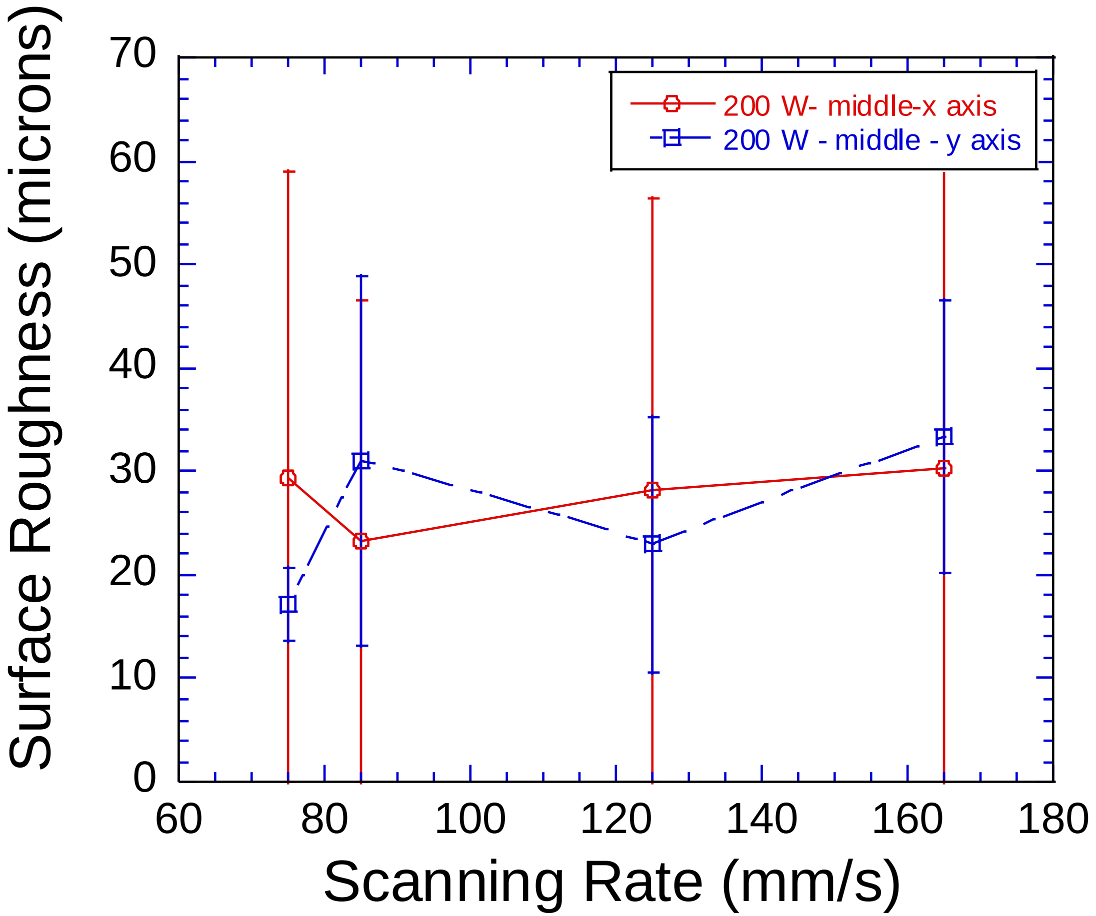
Ti-p200v75M Post-Clean



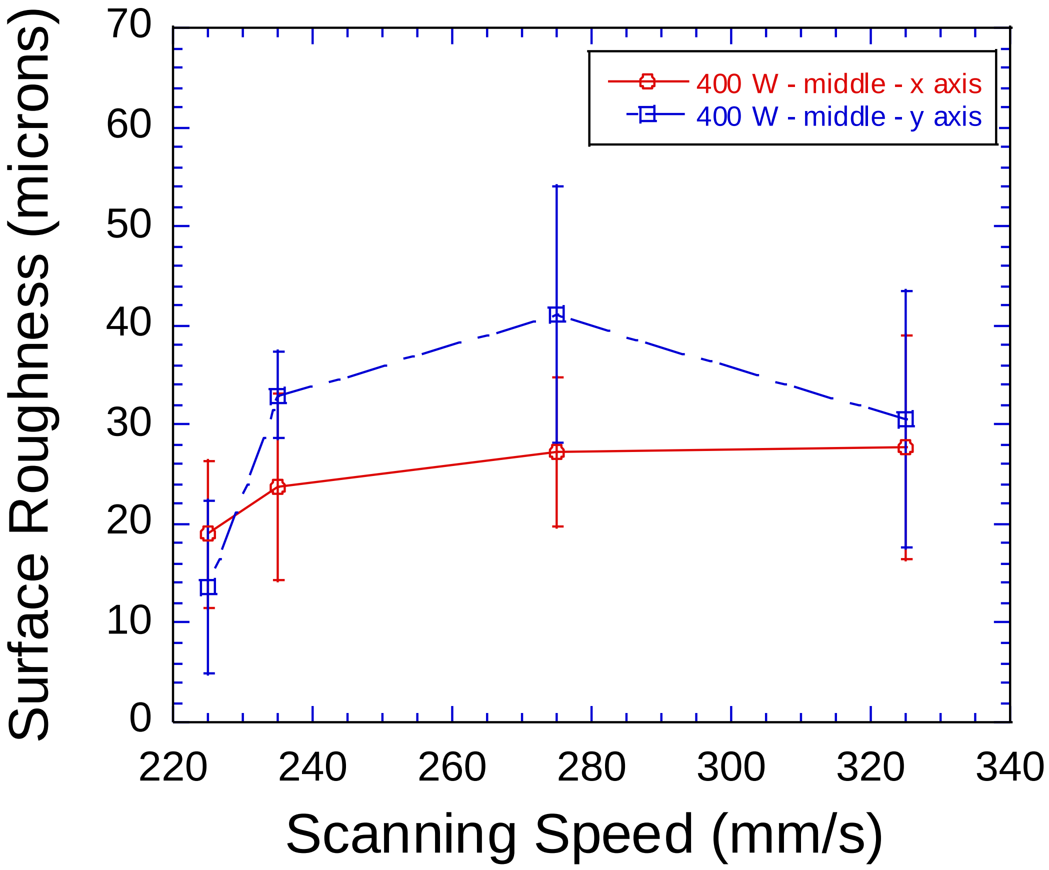
Profilometry Results: MAB on Ti Plate 100 Watts Post-Cleaning



Profilometry Results: MAB on Ti Plate 200 Watts Post-Cleaning



Profilometry Results: MAB on Ti Plate 400 Watts Post-Cleaning



Analysis/Summary

- ❖Based off the profilometry, higher wattages and slower scanning speeds appear to give lower relative surface roughness after sonication
- ❖Sonication at 60 minutes caused minor crack propagation
- ❖The SEM shows minimum decomposition at 100 watts
- ❖200 and 400 watts decompose the MAB more extensively

Next Steps/Outlook/Future Work

- If we had 4 more weeks, we would do tribological testing large section of 3D printed MAB phase
  - Gather more SEM data
- With 4 more months, we would try 3D printing different MAB compositions such as FeAlB
- MAB phases have potential for many different space applications

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Acknowledgements

- Mackenzie Short, Maharshi Dey, Caleb Matzke
- ND Space Grant Consortium
- NASA EPSCOR
- NASA Space Act Agreement with NASA Langley

References

[1] Grossin, D., Montón, A., Navarrete-Segado, P., Özmen, E., Urruth, G., Maury, F., Maury, D., Frances, C., Tourbin, M., Lenormand, P., & Bertrand, G. (2021). A review of additive manufacturing of ceramics by powder bed selective laser processing (sintering / melting): Calcium phosphate, silicon carbide, zirconia, alumina, and their composites. *Open Ceramics*, *5*, 100073. https://doi.org/10.1016/j.oceram.2021.100073

[2] King, W. E., Barth, H. D., Castillo, V. M., Gallegos, G. F., Gibbs, J. W., Hahn, D. E., Kamath, C., & Rubenchik, A. M. (2014). Observation of keyhole-mode laser melting in laser powder-bed fusion additive manufacturing. *Journal of Materials Processing Technology*, *214*(12), 2915–2925. https://doi.org/10.1016/j.jmatprotec.2014.06.005

Appendix: Power Density Key

p100v75	**p100v25**	**p200v125**	**p200v75**	**p400v275**	**p400v225**	**p250v100**
p100v85	**p100v35**	**p200v135**	**p200v85**	**p400v285**	**p400v235**	**p225v100**
p100v95	**p100v45**	**p200v145**	**p200v95**	**p400v295**	**p400v245**	**p200v100**
p100v105	**p100v55**	**p200v155**	**p200v105**	**p400v305**	**p400v255**	**p175v100**
p100v115	**p100v65**	**p200v165**	**p200v115**	**p400v315**	**p400v265**	**p150v100**
p100v125	**p100v75**	**p200v175**	**p200v125**	**p400v325**	**p400v275**	**p125v100**

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Appendix: Profilometry Results of MAB on Steel Plate Pre-Cleaning

#	p100v75L	p100v75M	p100v75R	p100v25L	p100v25M	p100v25R	p200v125L	p200v125M	p200v125R	p200v75L	p200v75M	p200v75R	p400v275L	p400v275M	p400v275R	p400v225L	p400v225M	p400v225R	p250v100L	p250v100M	p250v100R
x-axis	3.496	19.067	28.584	2.146	28.104	35.861	1.333	42.683	26.293	1.591	19.513	19.724	2.454	15.09	14.08	1.827	21.349	16.57			
y-axis	1.873	36.551	11.591	1.973	15.605	14.705	1.26	29.916	25.541	0.992	18.586	15.527	1.609	24.924	18.915	1.322	17.731	19.925			
#	p100v85L	p100v85M	p100v85R	p100v35L	p100v35M	p100v35R	p200v135L	p200v135M	p200v135R	p200v85L	p200v85M	p200v85R	p400v285L	p400v285M	p400v285R	p400v235L	p400v235M	p400v235R	p225v100L	p225v100M	p225v100R
x-axis	1.576	43.083	39.178	1.84	49.036	32.701	2.558	29.258	43.356	1.691	33.374	30.573	1.807	32.863	30.852	2.139	12.356	39.71	1.703	10.737	28.562
y-axis	1.701	48.383	38.919	2.278	19.567	17.153	1.466	36.505	41.055	1.255	27.501	18.561	2.053	35.492	30.208	1.505	38.884	25.091	1.143	10.039	19.29
#	p100v95L	p100v95M	p100v95R	p100v45L	p100v45M	p100v45R	p200v145L	p200v145M	p200v145R	p200v95L	p200v95M	p200v95R	p400v295L	p400v295M	p400v295R	p400v245L	p400v245M	p400v245R	p200v100L	p200v100M	p200v100R
x-axis	1.263	47.526	45.607	1.474	42.376	33.127	2.099	54.051	57.763	1.976	46.165	36.613	1.771	46.2	39.602	1.637	35.676	37.488	1.533	20.664	30.754
y-axis	2.281	24.05	61.578	1.039	38.882	31.328	1.408	29.592	25.274	1.61	19.216	32.063	2.47	25.722	45.371	1.485	28.602	40.679	1.416	31.291	8.307
#	p100v105L	p100v105M	p100v105R	p100v55L	p100v55M	p100v55R	p200v155L	p200v155M	p200v155R	p200v105L	p200v105M	p200v105R	p400v305L	p400v305M	p400v305R	p400v255L	p400v255M	p400v255R	p175v100L	p175v100M	p175v100R
x-axis	3.182	51.993	36.935	2.43	37.063	30.288	2.451	30.889	56.556	1.37	23.787	36.507	2.09	27.292	41.388	2.203	22.699	16.31	1.55	26.891	30.959
y-axis	4.922	40.045	37.681	2.76	31.266	21.609	2.472	44.273	34.399	1.876	34.506	41.286	1.281	25.651	24.696	1.939	47.915	37.573	1.727	23.453	40.051
#	p100v115L	p100v115M	P100v115R	p100v65L	p100v65M	p100v65R	p200v165L	p200v165M	p200v165R	p200v115L	p200v115M	p200v115R	p400v315L	p400v315M	p400v315R	p400v265L	p400v265M	p400v265R	p150v100L	p150v100M	p150v100R
x-axis				2.267	21.261	37.759	2.924	40.948	24.79	1.64	64.82	28.36	2.036	24.407	21.087	1.26	61.67	43.474	2.107	54.694	44.82
y-axis				1.016	21.792	38.547	1.522	29.567	38.863	0.753	27.154	30.481	1.668	23.013	55.264	1.133	27.092	29.022	2.79	24.153	32.363
#	p100v125L	p100v125M	p100v125R	p100v75L	p100v75M	p100v75R	p200v175L	p200v175M	p200v175R	p200v125L	p200v125M	p200v125R	p400v325L	p400v325M	p400v325R	p400v275L	p400v275M	p400v275R	p125v100L	p125v100M	p125v100R
x-axis							2.056	32.406	37.608	1.78	21.734	22.733	1.538	18.934	18.879						
y-axis							2.031	40.921	25.663	2.092	19.427	21.419	1.552	34.198	34.79						

Appendix: Profilometry Results of MAB on Ti Plate Pre-Cleaning

#	p100v75L	p100v75M	p100v75R	p100v25L	p100v25M	p100v25R	p200v125L	p200v125M	p200v125R	p200v75L	p200v75M	p200v75R	p400v275L	p400v275M	p400v275R	p400v225L	p400v225M	p400v225R	p250v100L	p250v100M	p250v100R
x-axis	1.528	36.467	24.293	4.239	20.425	42.188	1.968	34.095	36.199	1.619	26.165	21.418	3.706	36.447	25.198	3.836	15.623	19.931	1.607	24.525	31.428
y-axis	0.836	19.047	19.649	0.507	27.724	18.743	1.347	30.149	37.201	1.413	15.857	15.45	2.697	42.696	31.982	1.57	20.666	13.359	1.306	15.1	46.318
#	p100v85L	p100v85M	p100v85R	p100v35L	p100v35M	p100v35R	p200v135L	p200v135M	p200v135R	p200v85L	p200v85M	p200v85R	p400v285L	p400v285M	p400v285R	p400v235L	p400v235M	p400v235R	p225v100L	p225v100M	p225v100R
x-axis	0.934	30.731	38.603	1.211	35.253	52.382	2.034	22.573	19.599	2.494	29.742	31.663	3.905	12.123	44.331	4.571	24.354	52.554	1.714	40.692	39.6
y-axis	0.675	17.023	35.163	0.419	40.532	54.689	1.574	43.999	29.493	1.388	26.682	23.789	2.5	35.44	30.167	1.803	32.612	31.993	1.369	13.447	43.622
#	p100v95L	p100v95M	p100v95R	p100v45L	p100v45M	p100v45R	p200v145L	p200v145M	p200v145R	p200v95L	p200v95M	p200v95R	p400v295L	p400v295M	p400v295R	p400v245L	p400v245M	p400v245R	p200v100L	p200v100M	p200v100R
x-axis	0.808	47.78	52.382	0.684	40.313	37.311	1.902	54.258	21.535	2.12	37.605	46.044	1.648	36.268	26.514	1.626	24.848	47.641	1.936	15.033	46.689
y-axis	0.845	46.382	32.791	0.404	45.362	33.781	1.534	33.991	29.722	1.26	37.17	26.622	1.392	22.113	16.033	1.665	12.622	31.499	1.368	10.947	40.291
#	p100v105L	p100v105M	p100v105R	p100v55L	p100v55M	p100v55R	p200v155L	p200v155M	p200v155R	p200v105L	p200v105M	p200v105R	p400v305L	p400v305M	p400v305R	p400v255L	p400v255M	p400v255R	p175v100L	p175v100M	p175v100R
x-axis				1.647	42.515	31.214	1.585	42.077	26.905	1.351	23.241	28.523	1.718	29.854	29.847	1.956	19.542	11.732	1.138	34.465	36.385
y-axis				0.867	32.492	17.999	1.347	53.465	36.952	1.036	11.54	48.03	1.346	42.161	37.778	1.446	30.897	48.787	1.271	39.548	28.08
#	p100v115L	p100v115M	P100v115R	p100v65L	p100v65M	p100v65R	p200v165L	p200v165M	p200v165R	p200v115L	p200v115M	p200v115R	p400v315L	p400v315M	p400v315R	p400v265L	p400v265M	p400v265R	p150v100L	p150v100M	p150v100R
x-axis				1.035	29.325	45.124	2.801	49.047	28.461	2.156	51.219	26.016				1.984	41.247	43.337	1.363	24.148	30.221
y-axis				0.786	33.277	39.25	1.558	27.993	34.464	1.456	23.162	19.795				1.568	41.876	26.243	0.876	31.586	23.719
#	p100v125L	p100v125M	p100v125R	p100v75L	p100v75M	p100v75R	p200v175L	p200v175M	p200v175R	p200v125L	p200v125M	p200v125R	p400v325L	p400v325M	p400v325R	p400v275L	p400v275M	p400v275R	p125v100L	p125v100M	p125v100R
x-axis										2.254	18.767	43.035	2.173	46.886	30.615	2.741	28.629	19.916	0.767	50.55	33.614
y-axis										1.367	8.154	28.671	1.461	57.914	42.989	1.466	36.572	39.651	0.37	38.999	30.641

Abstract

During the ten-week internship my work focused on designing characterization protocol for 3D printed MAB phase ceramics on steel and Ti plates. I was mentored by Dr. Samuel Hocker of NASA and advised by Dr. Gupta from University of North Dakota. I also collaborated with Mackenzie Short from the University of North Dakota.

The characterization protocol was divided into following steps; initially, we performed visual inspection of the plates. During this process, the coatings which spalled or delaminated were discarded for further evaluation. During the next step, we performed optical microscopy to discern and document surface features and porosities of the samples. We also noted some decomposition under the optical microscope as well. Thereafter, profilometry analysis was performed to understand the effect of power density on the surface roughness. Due to the deposited powder over the samples, the profilometry was inconclusive. We then cleaned the samples on the steel plate using ethanol and cotton swabs to wipe up the loose powder covering the samples. Detailed SEM analysis was then performed on the cleaned specimens. In parallel, the 3D printed samples on the Ti plate were cleaned by using an ultrasonic bath for 1h. Optical imaging showed that this cleaning process was more effective for cleaning except that small cracks propagated through the layered ceramics. The profilometry data gathered on the titanium plate after cleaning was more conclusive. The SEM data on the steel plate showed that there is decomposition at all wattages although at 100 W the extent of decomposition was lower. We are planning to characterize the steel samples after cleaning in ultrasonic bath for 5-10 min to minimize cracking during the cleaning process. In addition, we are also planning to study the fractured surface by SEM analysis.