Ablation Evaluation
May 2021
SN1535

LIGO-T2100216

Billingsley, Zhang

Advanced LIGO Core Optic coating witness sample ETM 07/09

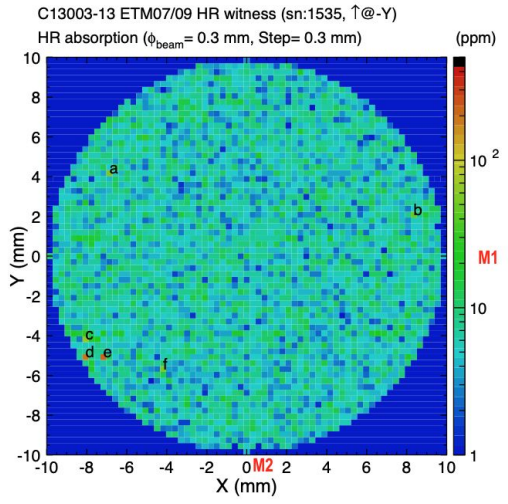
SN1535 main DCC card at LIGO-E2000023

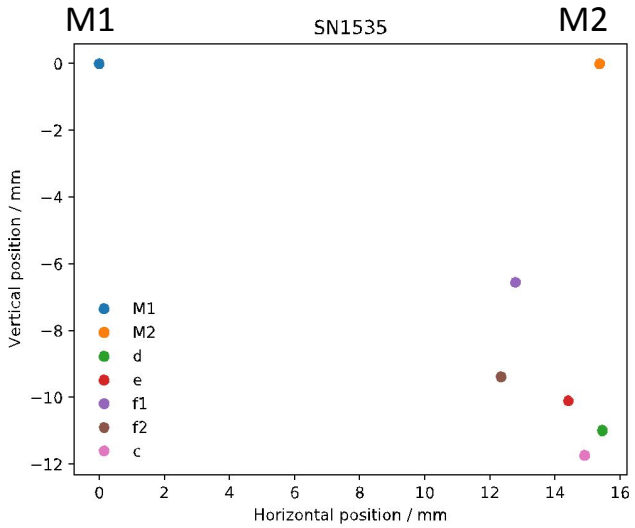
Initial absorption scan also at LIGO-E2000023

Pre-ablation SEM images at LIGO-G2100805-v1 (Page 4 contains MIT's correctly oriented SEM data)

Key + History + Timeline (Updated 2021.11.01)

CIT RTS measurement established presence of absorbers
MIT SEM measurement was in correct orientation, found Al in one feature in region D (G2100805 pgs 4 and 5)
MIT ablation effort introduced an error in orientation, and as a result ablation regions are not colocated with CIT absorbers.
CIT SEM follow up found that region D, and the aluminum feature within region D, had not been ablated. (same for ablations in F)
CIT RTS follow up measurements have focused on the ablated features, not the originally found absorbers
Plasma clean (carbon) plus annealing at 300 C for 10 hours appear to reduce/remove absorption signal in ablated areas
Anneal to reduce/remove absorption signal in ablated areas
CIT RTS follow up Absorption scan confirmed anneal reduced/removed ablated spots
Send to MIT for ablation of known metallic - now named od near (-8,-5)

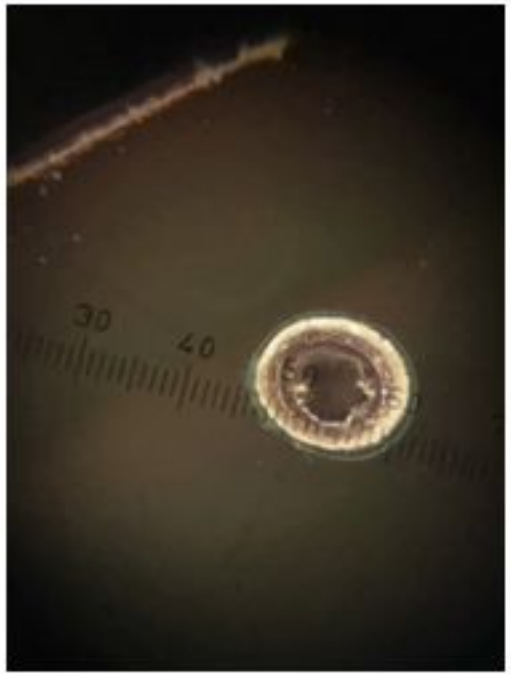


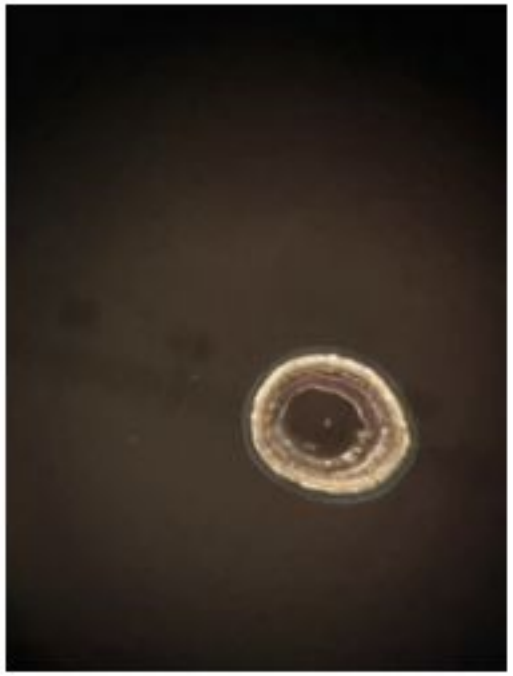


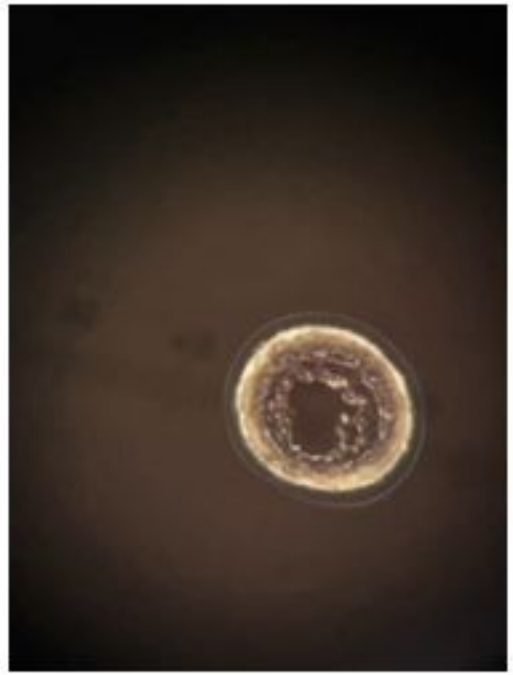
No	Absorption (ppm)	X_{RTS} (mm)	Y_{RTS} (mm)	X_{optic} (mm)	Y_{optic} (mm)	Comments
a	59	63.691	61.625	-6.309	6.375	
b	49	78.582	65.404	8.582	2.596	
c	69	62.686	70.393	-7.314	-2.393	
d	192	62.816	71.292	-7.184	-3.292	
e	223	64.181	71.201	-5.819	-3.201	
f1	59	67.823	72.619	-2.177	-4.619	
f2	50	66.158	70.287	-3.842	-2.287	New found
M1		81.553	68.407	11.553	-0.407	
M2		70.538	79.119	0.538	-11.119	Next to arrow

Arrow at bottom-M2

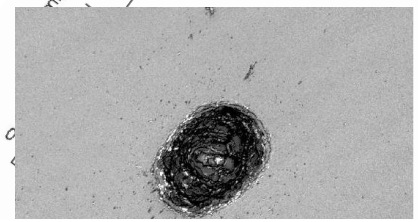
There are 3 fiducial marks on SN1535

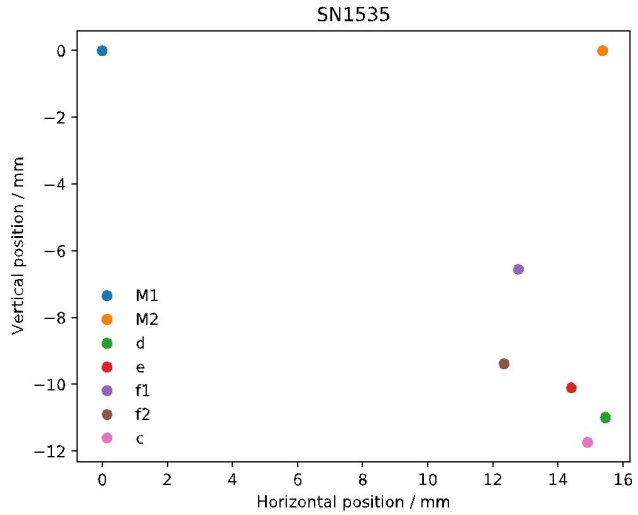




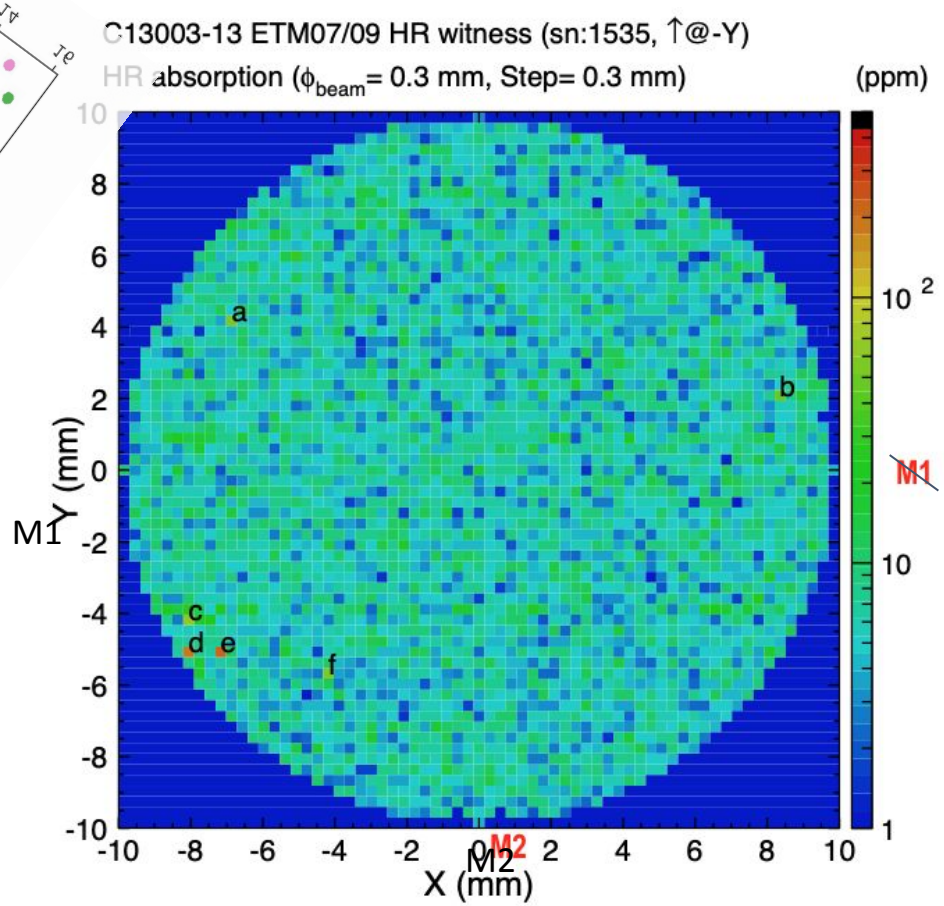


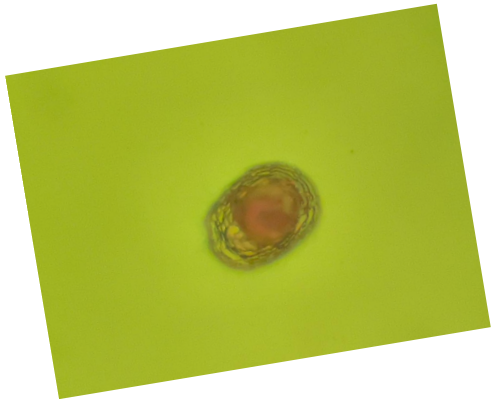
Found new spot D as described with the following orientation, measured 36µm x 26µm.

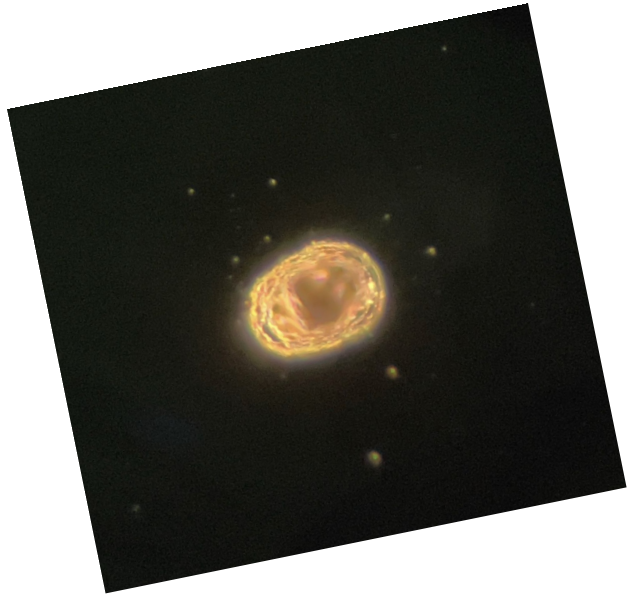




SN1535-MIT Coord		
Region	X / mm	Y / mm
M1	0.00	0.00
M2	15.36	0.00
d	15.44	-10.99
e	14.40	-10.11
f1	12.78	-6.55
f2	12.35	-9.39
c	14.91	-11.73



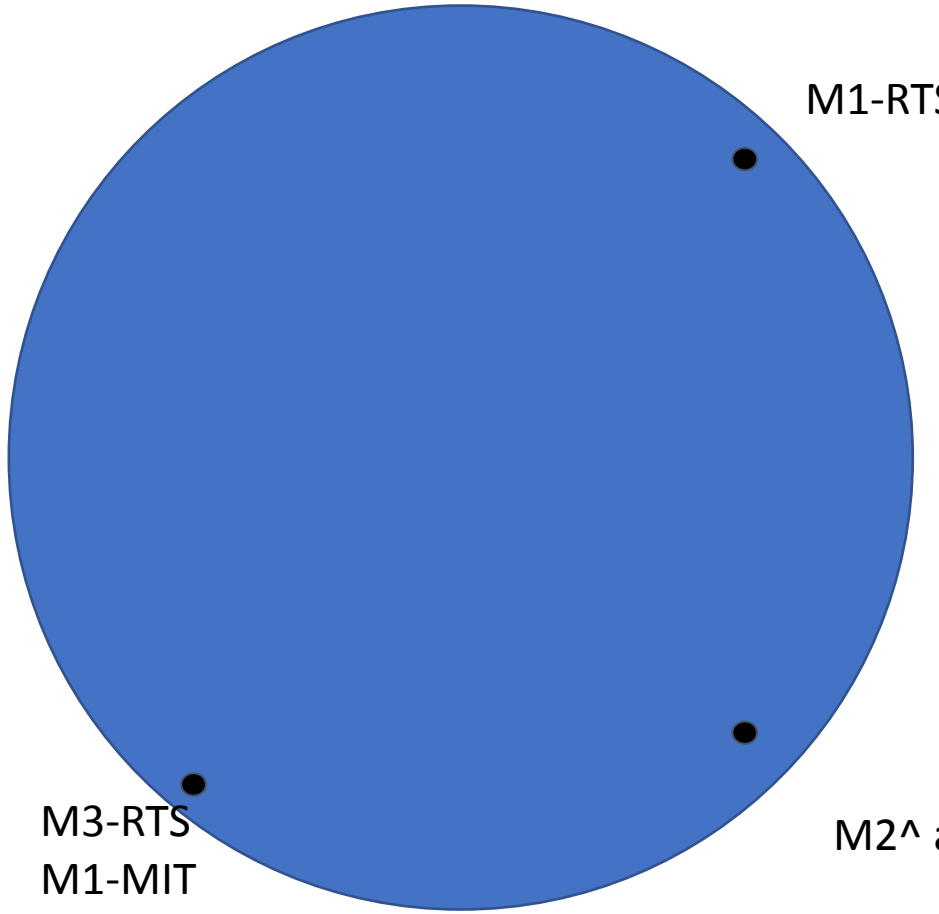




Arrow at bottom-M2

Different coordinate system than in the absorption scan found at E2000023 ...

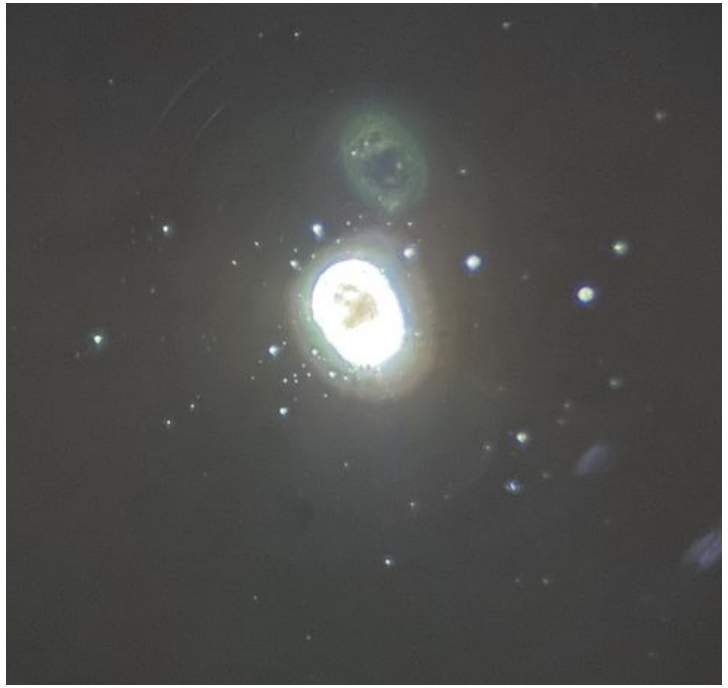
There are three fiducial marks on SN1535. There are two coordinate systems. The confusion stems from which mark is called M1

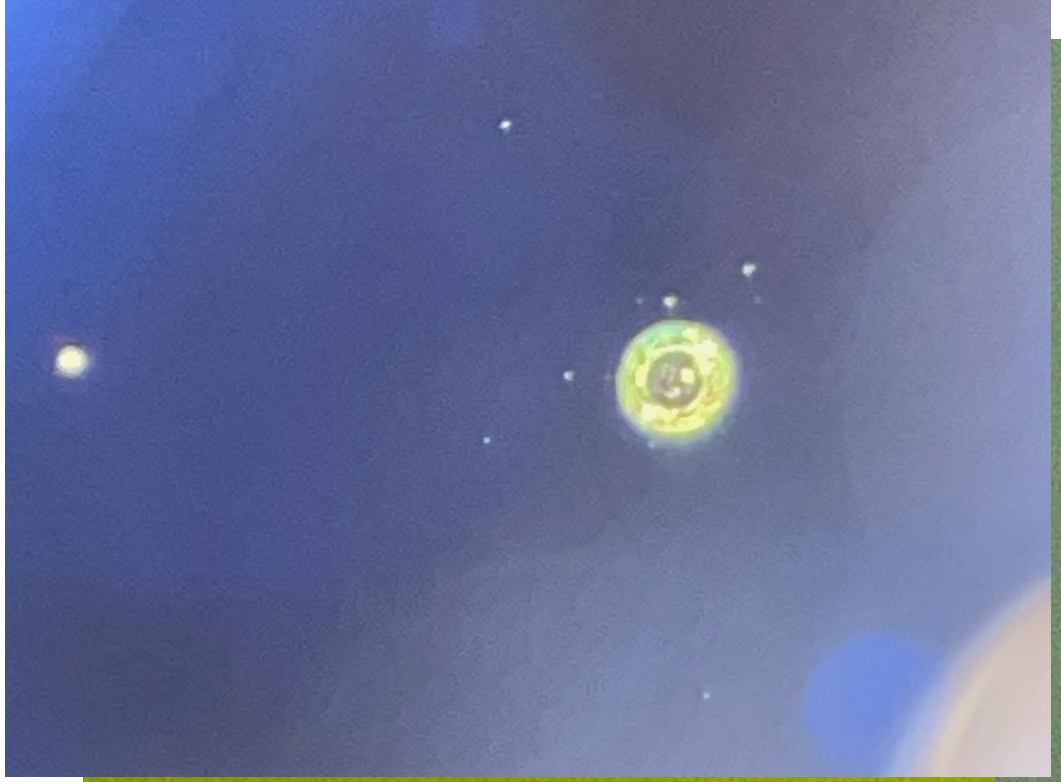


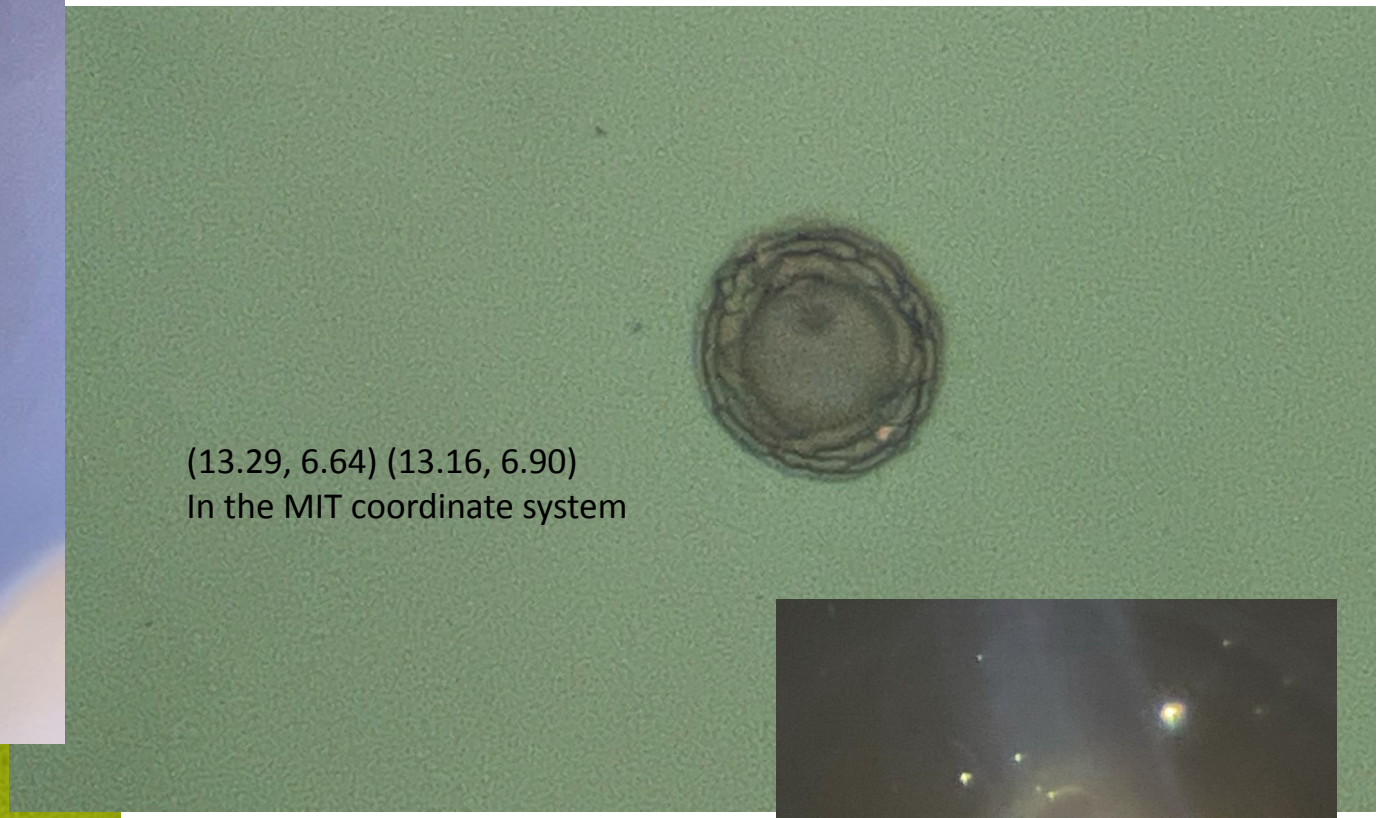
SN1535-RTS Coord Ablation spots found by microscope at the following locations			
Region	X / mm	Y / mm	
M3	0.00	0.00	used by MIT as M1
M2	15.66	0.00	
M1	16.45	15.32	
d	14.90	10.50	
f1a	13.29	6.64	
f1b	13.16	6.90	

Before/After 1h 40m ultrasonic clean – difference could be explained by exposure

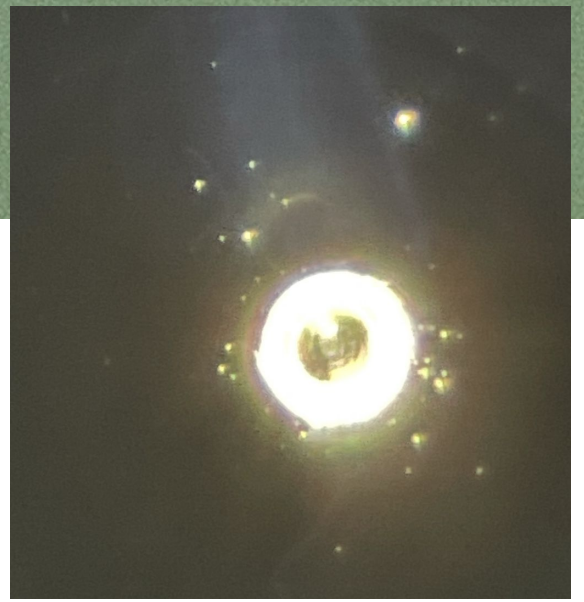
- Ultrasonic clean in acetone for 1:40
- Spray with IPA
- Drag wipe with acetone

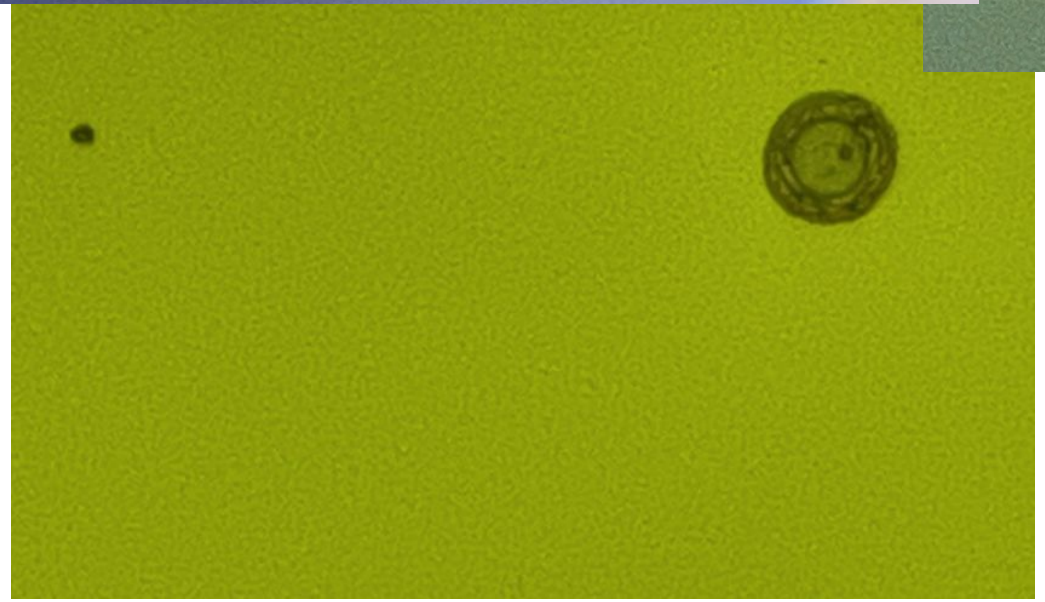






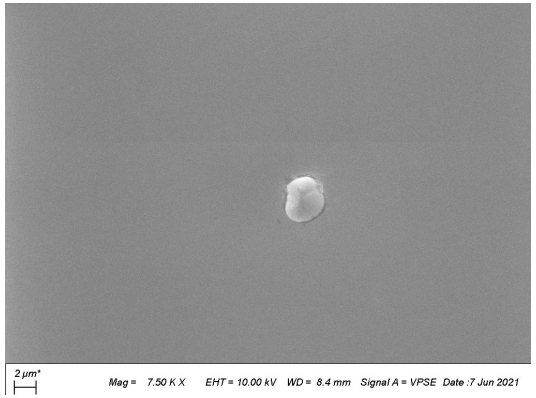
(13.29, 6.64) (13.16, 6.90)
In the MIT coordinate system



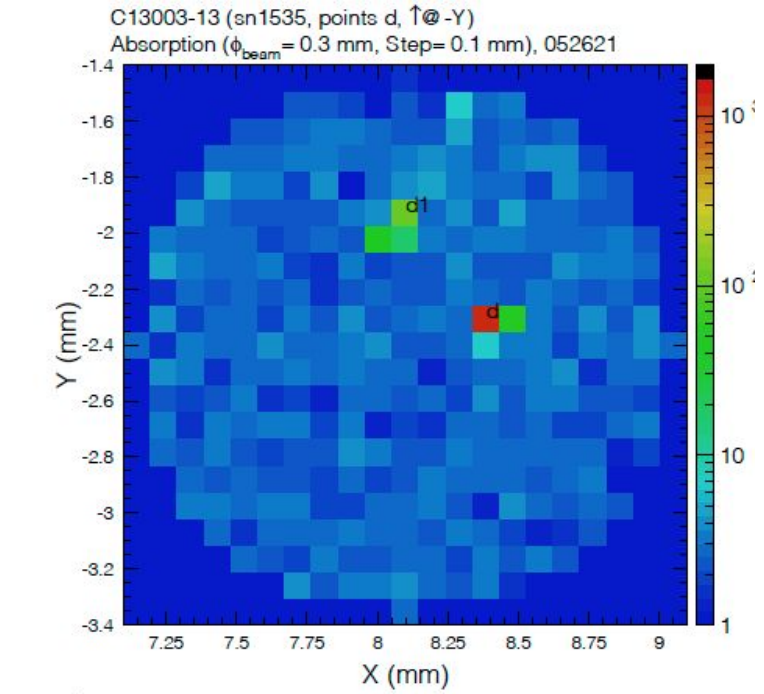


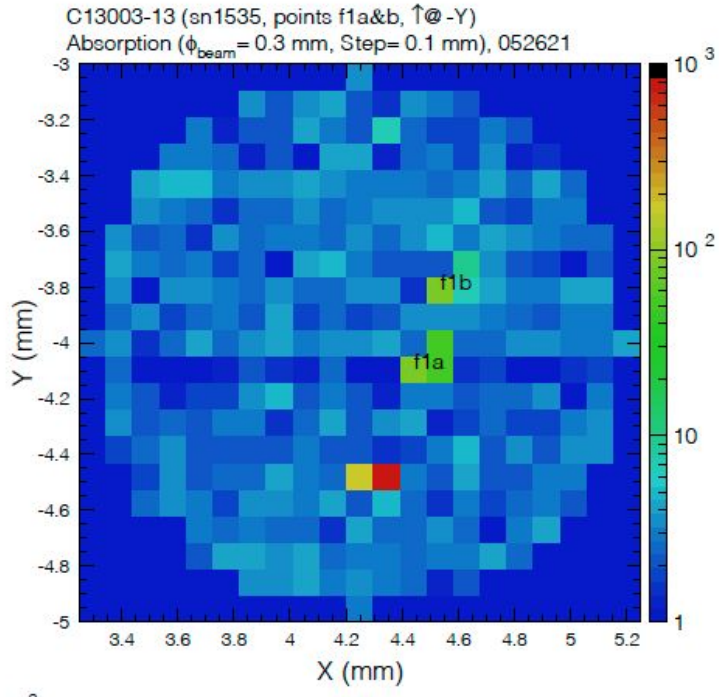
High resolution absorption scan post-ablation

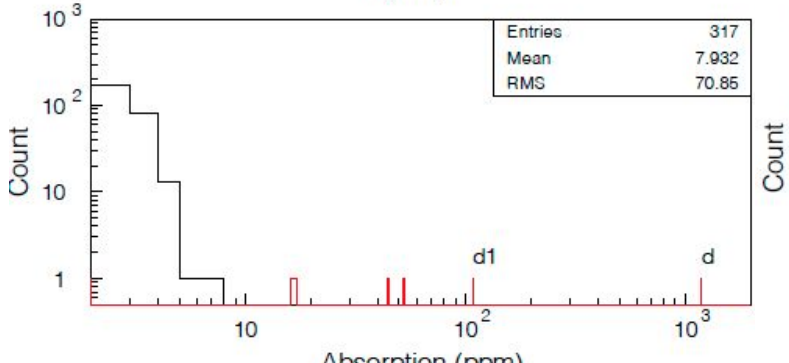
Liyuan found the mystery spot near f1a, it is a ~5 µm dark spot, filed with this document at LIGO-T2100216

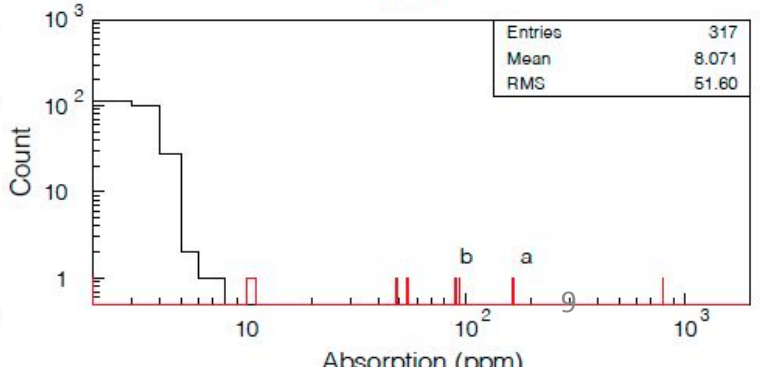


SEM Image of the apparent absorbing feature in region F.
ref. image SN1535_f1c_01.tif







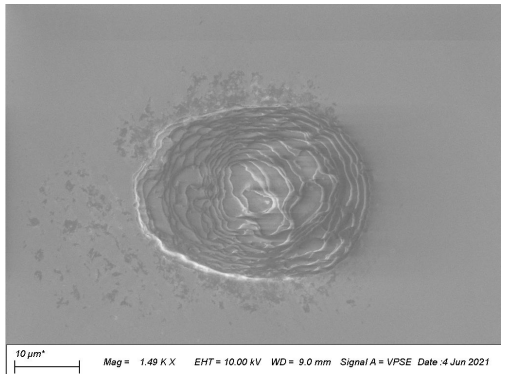


SEM EDS spectra post-ablation - Feature D

Imaging used to correctly identify M1, M2, and M3, leading to establishment of coordinate system for easy feature location.

No Aluminum identified in most spectra measured near Feature D on SN1535 (or near Feature F, either).

Two solo imaging sessions included similar findings - WIP to confirm this absence of Aluminum with guidance from SEM expert.



Feature D - image ref. SN1535_d_01.tif

Typical spectrum comparing D (feature) to D1 (test ablation of clean coating) highlights absence of Al.

SEM EDS spectra post-ablation - Feature D

Some details / possible snags:

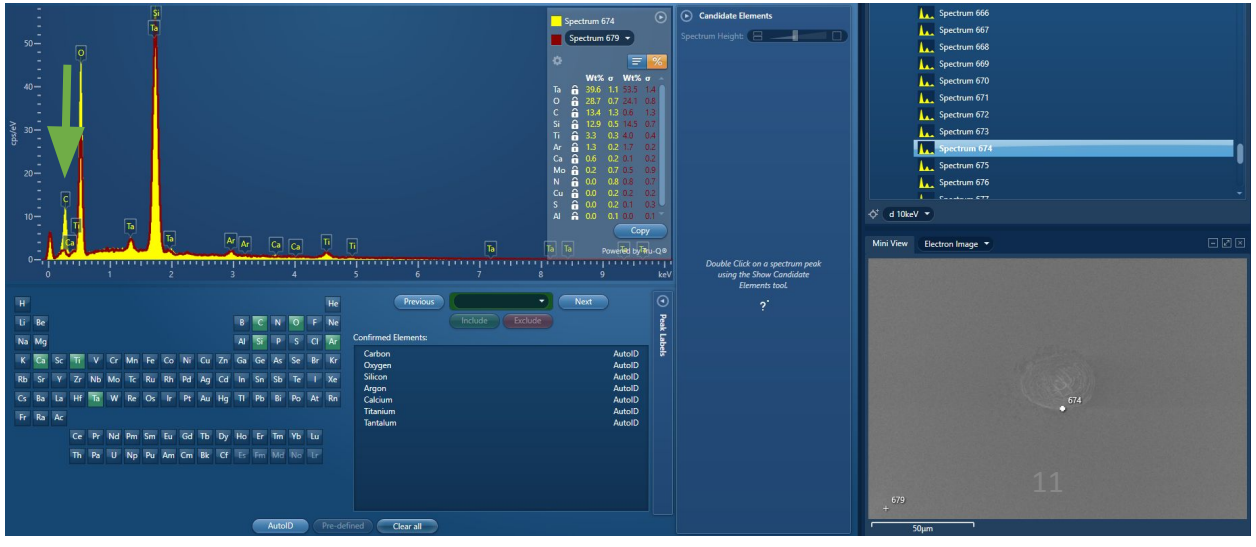
(right) trace of aluminum present in one spectrum, on level similar to error term.

(bottom) bumps of C and apparently Na, and maybe P and Ca, in one area.

I want to work with Chi to confirm and better understand these features.

0.1% Aluminum (sigma 0.1%) in this particular point spectrum - is this meaningful?

small spike in C at nearby locations, not present in the rest of the spectra throughout the region - meaningful? also appears to include elevated Na, Ca, maybe P (though I think it could be misidentified Ta)



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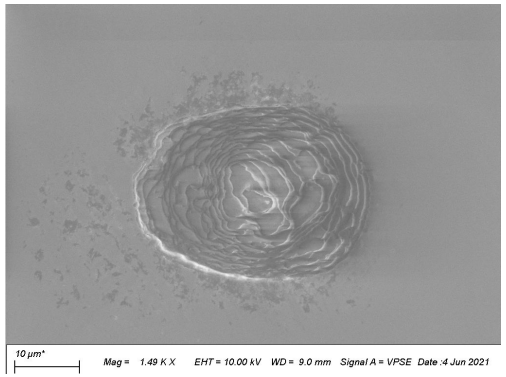


Feature D - image ref. SN1535_d_01.tif

Typical spectrum comparing D (feature) to D1 (test ablation of clean coating) highlights absence of Al.

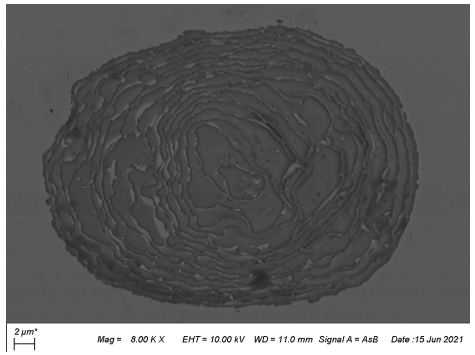
Update 18 June 2021

SEM EDS spectra post-ablation - Feature D

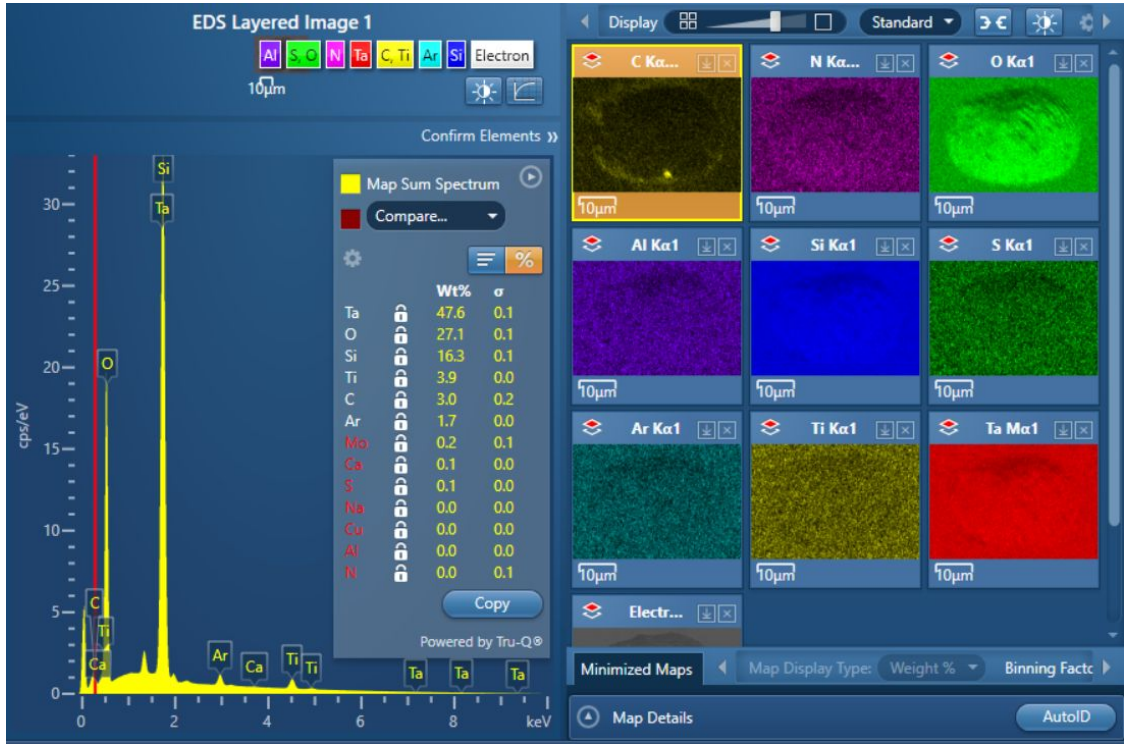
Right: High resolution mapping shows presence of Carbon. Yellow map shows Carbon signal which appears to follow the border uniformly except for one local subregion with greater signal, at 6 o'clock. This imaging does not indicate any trace metals of interest or any other signatures. Main ablation area composition is similar to coating.

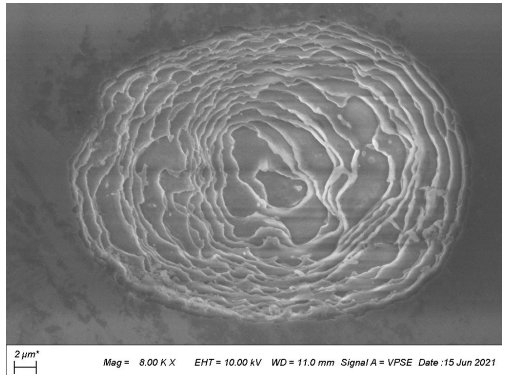
(note shadowing effect is due to detector mounting off-axis at 12 o-clock position - indicates feature falls below surrounding surface, and artificially suppresses signal at 12 o'clock position)

Need to image "clean coating" ablations to compare and understand differences. - work planned for Tuesday 29 June.



Feature D - Backscatter detector shows contrasting subregion at 6 o'clock at border. Also, uniform contrast suggests we may not have reached substrate.
image ref. SN1535_d_21.tif





Feature D - standard detector, horizontal artifact lines due to charging at edges. image shows array of edges (layers?) (why apparently more than 8, per standard AR stack)
Image ref. SN1535_d_22.tif

SEM EDS spectra post-ablation - Feature F1C

Ref. Slide 8 - apparent high absorber near clean-coating-ablation features F1A and F1B.

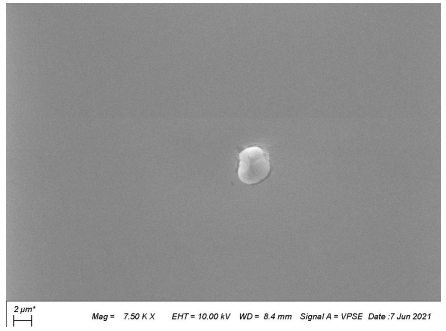
Top Right - spectrum showing typical scan, at center of 4 micron diameter feature. Red trace for comparison is adjacent “clean coating”. No signature of interest.

Bottom Right - spectrum showing localized Carbon peak (7.6% wt. compared to 1.3% wt.).

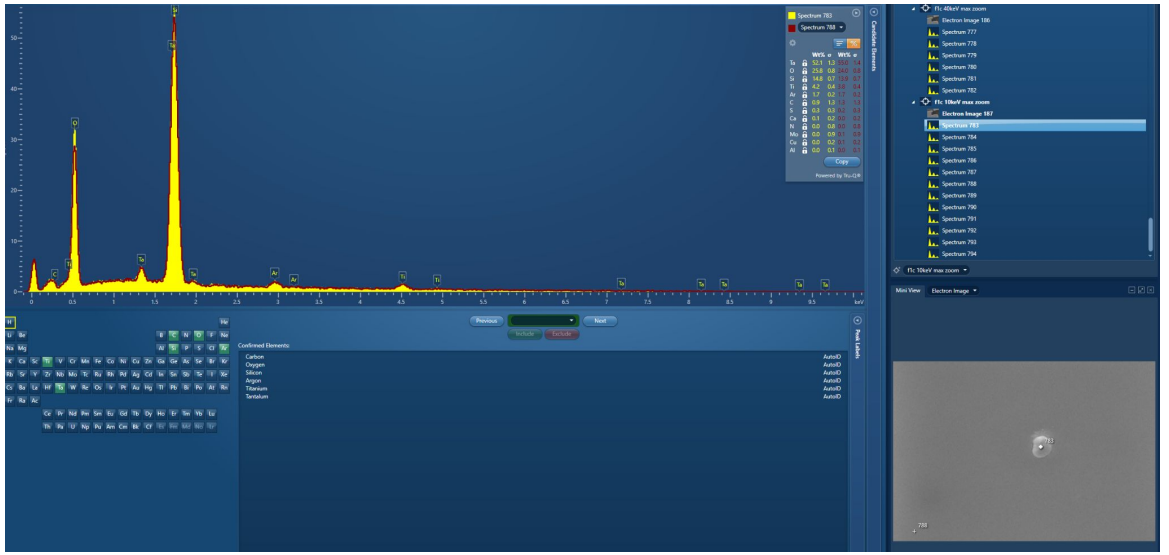
Localized Carbon signature warrants additional backscatter imaging to try to understand the extents of a potential subregion including Carbon.

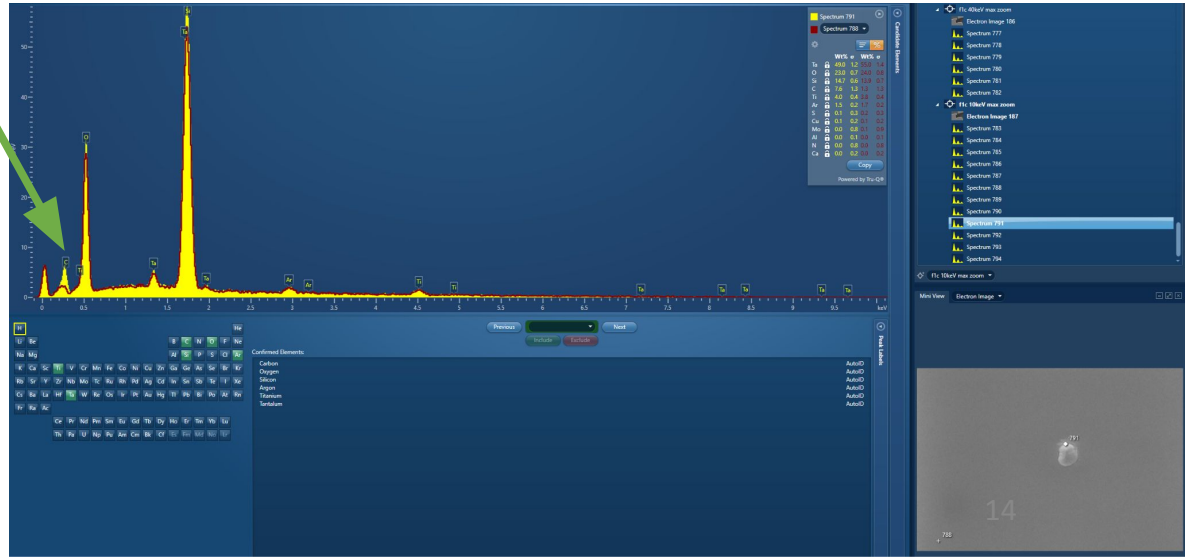
Update: imaging complete

on next slide.



SEM Image of the apparent absorbing feature in region F.
ref. image SN1535_f1c_01.tif





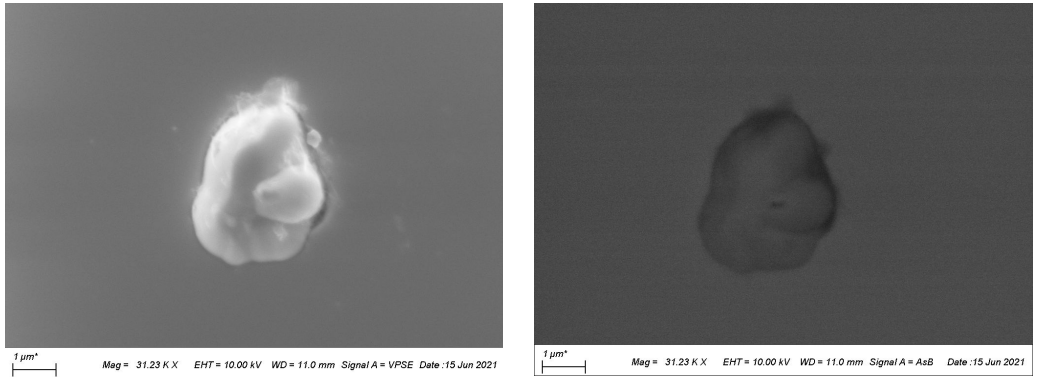
Update 18 June 2021

SEM EDS spectra post-ablation - Feature F1C

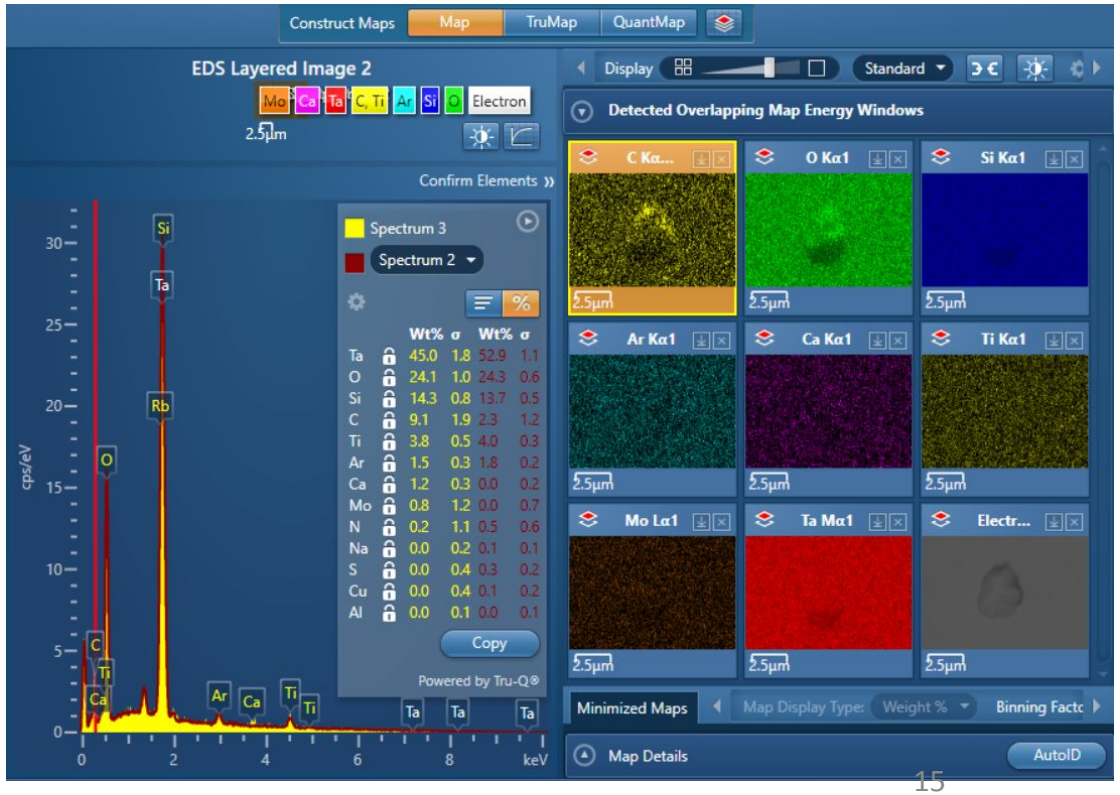
Right: High resolution mapping shows presence of Carbon. This imaging does not indicate any trace metals of interest or any other signatures. Main particle composition is similar to coating.

(note shadowing effect is due to detector mounting off-axis at 12 o-clock position - indicates feature protrudes above surrounding surface, and artificially suppresses signal at 6 o'clock position)

Below: Presence of contrasting material (such as Carbon) is indicated in Backscatter detector image around the border, consistent with map findings. Main particle seems to have composition similar to coating, based on contrast of BS image.



Feature F1C - SEM Images of the apparent absorbing feature in region F.
Left: VPSE detector (standard) Right: Backscatter detector
ref. image SN1535_f1c_22.tif and image SN1535_f1c_22.tif



Update 29 June 2021

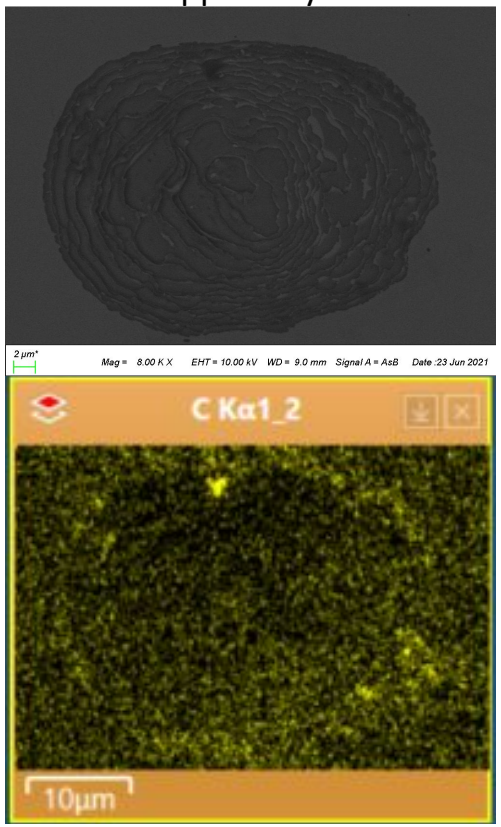
SEM EDS spectra post-ablation - all ablations

High resolution mapping shows presence of Carbon present in all ablations, around the border.

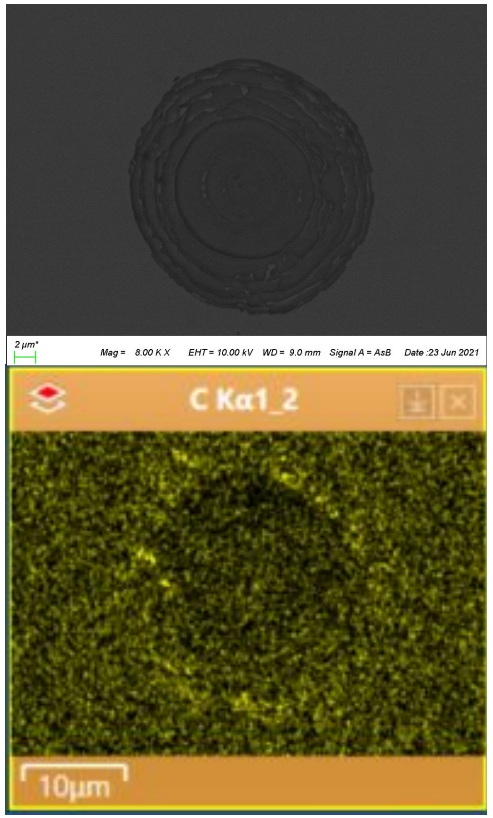
Carbon is more prominent in Feature D (including strong localized feature). Current speculation is that this could be related to conductive polymer coating applied by MIT. These results are consistent with this speculation, as all features show pretty similar C signature.

SEM Backscatter Detector

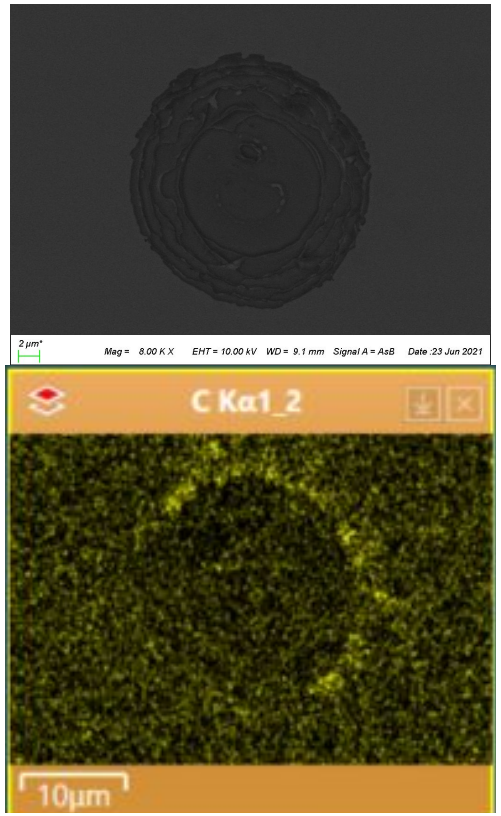
EDS Carbon Map



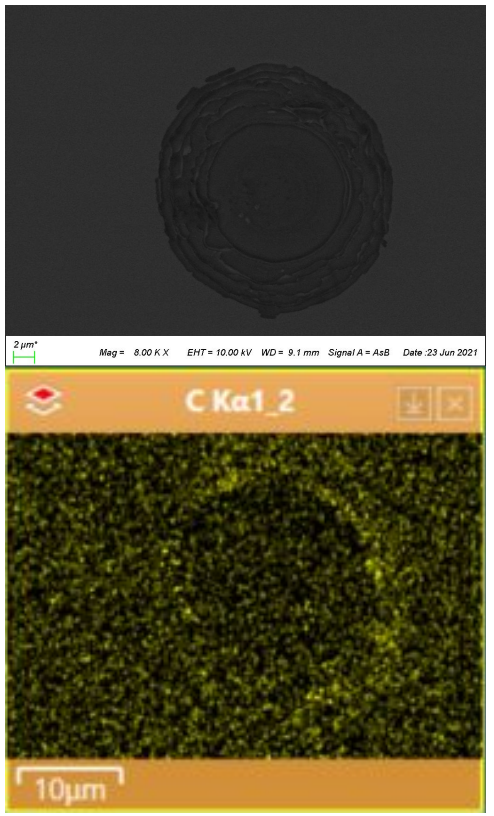
D
2.6% C, sum over area



D1
(clean coating)
2.1% C sum



F1A
2.2% C sum



F1B
2.2% C sum

Note that images are rotated 180° from typical mounting, I was interested in comparing shadow effect and signal levels. This check found no issues.

Update 07 July 2021

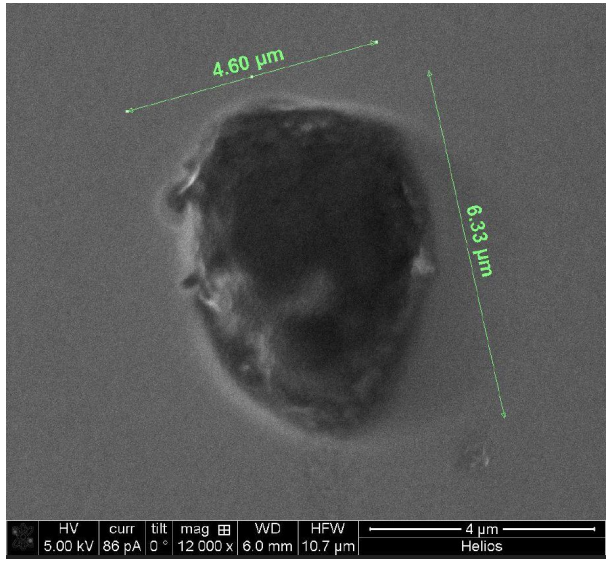
Feature D lives on, we ablated something else

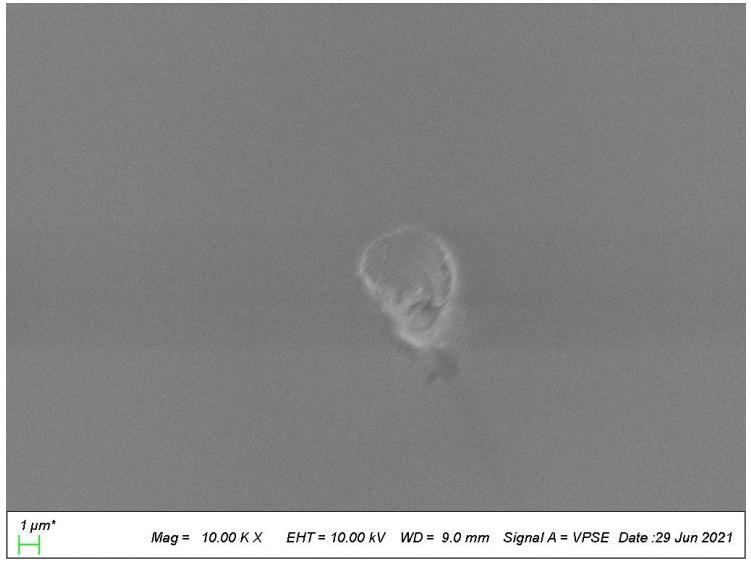
Looks like some of the confusion around coordinate systems led to ablation of something different from Feature D.

Stephen used the RTS coordinate system to navigate to the location of Feature D, and it turns out that Feature D is still present on the surface of this optic.

We will proceed with the slated tests (plasma cleaning to try to remove carbon signature from previous slide).

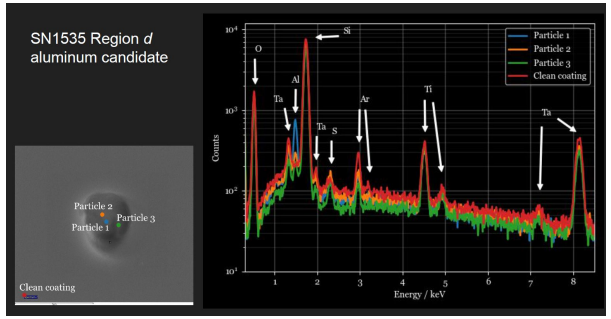
D from Kevin's https://dcc.ligo.org/LIGO-G2100805 (slide 4)

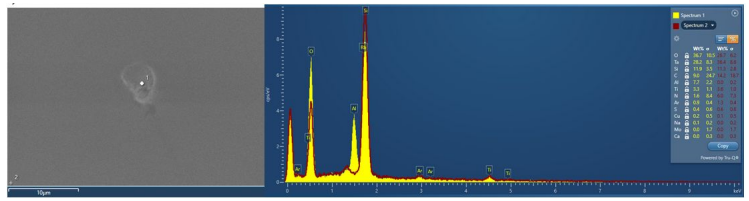




D from Stephen's https://dcc.ligo.org/T2000733 - comparable feature size and shape (ref. sn1535_d_52.tif)

Kevin found local, small aluminum signal (ref. his slide 5)





Stephen's found the aluminum signal (data not posted yet)

Update 12 July 2021

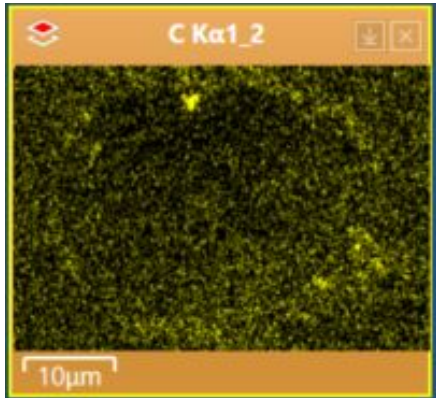
O_2 Plasma Cleaning removed Carbon concentrations

High resolution mapping showed presence of Carbon in all ablations, concentrated around borders. Beyond curiosity about the source, this prompted 2 questions: 1) is it possible to remove this C concentration? 2) is the C responsible for absorption around the ablations found in RTS measurements?

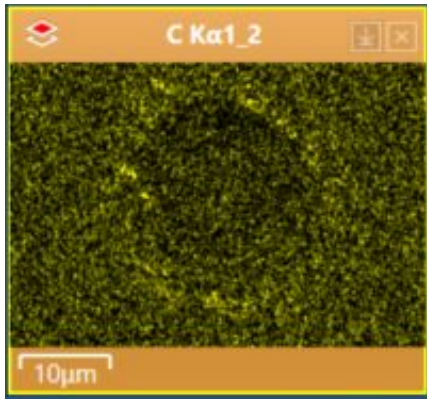
To address 1), the witness sample was cleaned using an O_2 plasma. After, the concentrations of carbon were no longer present, with C levels reduced and now on par with "clean coating" areas. Absorption scan next, to attempt to address question 2).

EDS Carbon Maps **(before clean)**

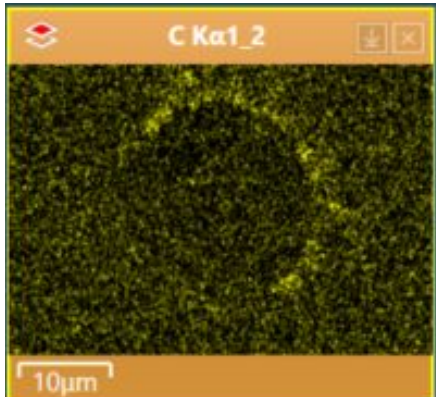
ref. sn1535 20210623 bs.oip



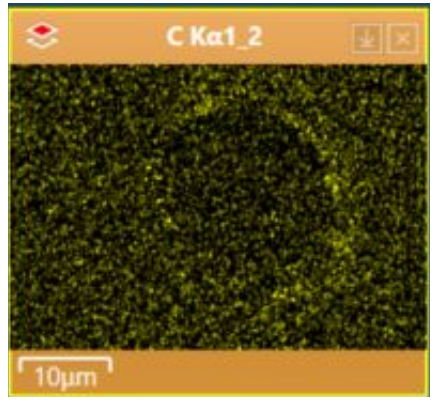
D
2.6% C (0.3%) before
→ 3.0% (1.6%) (larger error, smaller peak by eye)



D1 (clean coating)
2.1% (0.2%) before
→ 1.8% (0.7%)



F1A
2.2% (0.3%) before
→ 1.6% (0.5%)

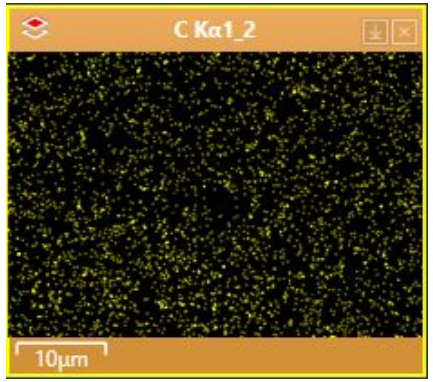


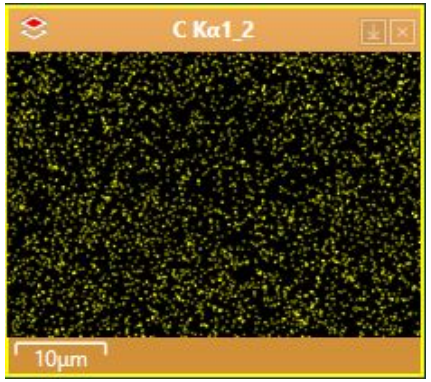
F1B
2.2% (0.3%) before
→ 1.6% (0.6%)

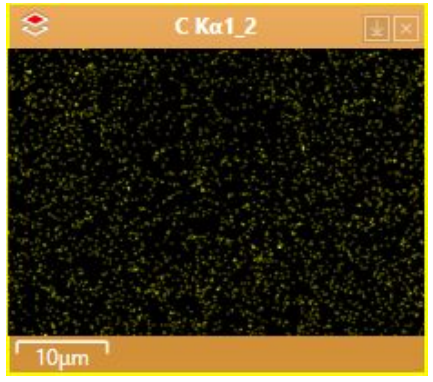
Note that images are rotated 180° from typical mounting, I was interested in comparing shadow effect and signal levels. This check found no issues.

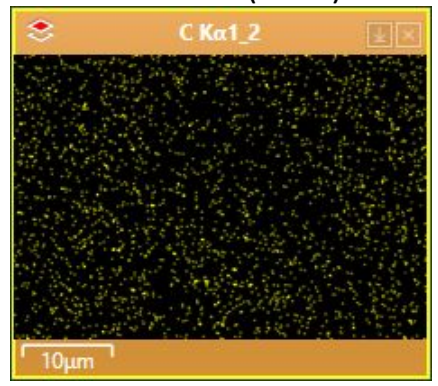
EDS Carbon Maps **(after clean)**

ref. sn1535 after plasma clean 20210709.oip





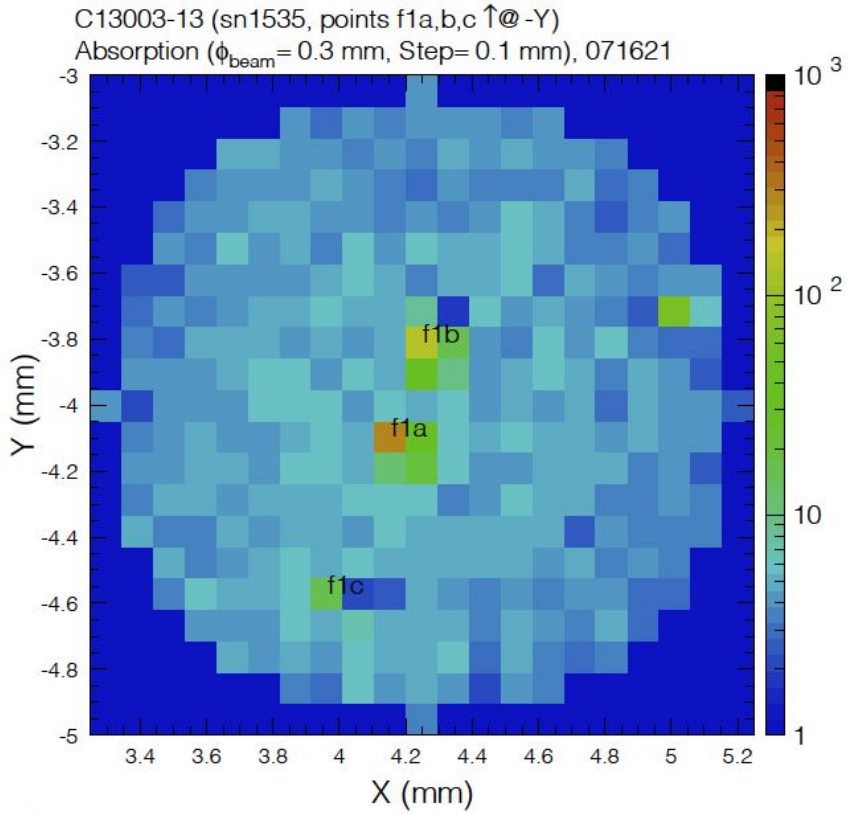


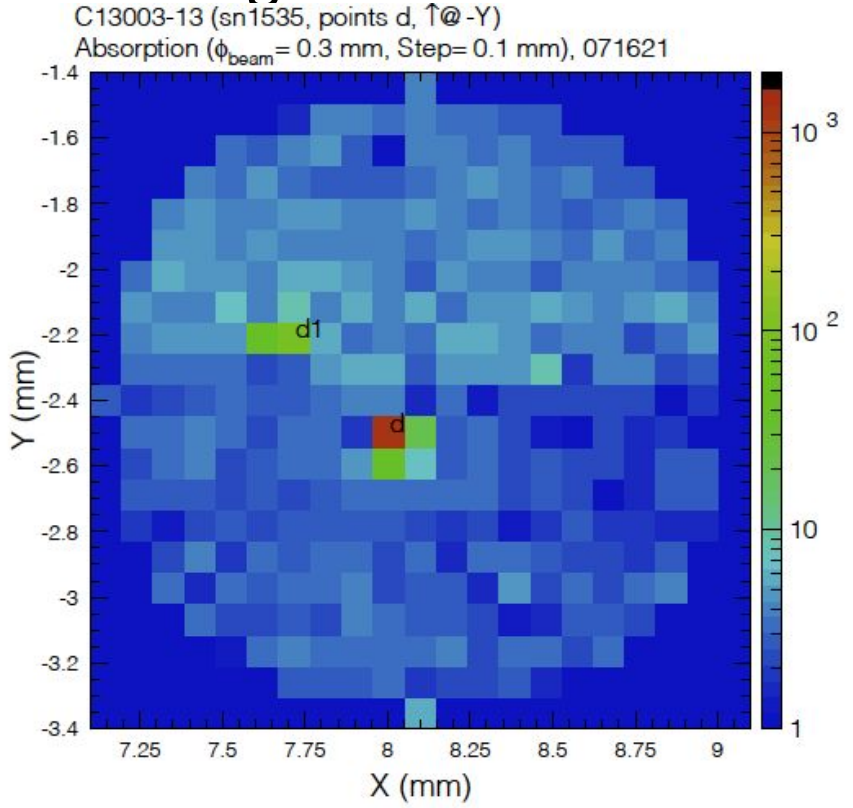


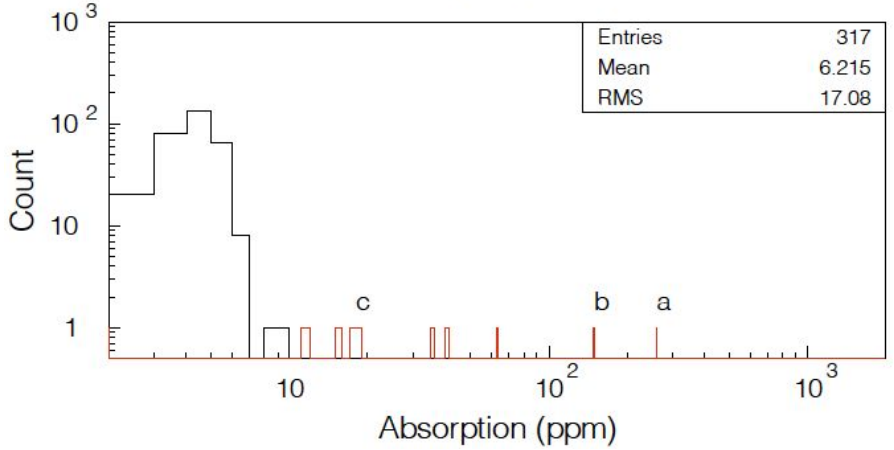
Absorption unchanged for ablated spots after plasma cleaning

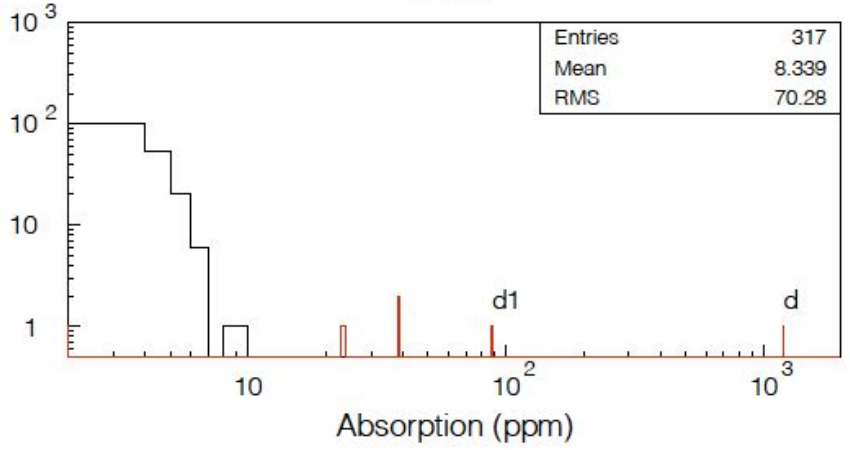
Absorption at f1c is significantly reduced.

Absorption at all ablated spots remains the same.









HWS Point absorber measurement - SN1535

See a similar point absorber at same location new-“d” as RTS [+8, -2.5]mm

31-Aug-21: testing original point “d” at [-8, -5]mm

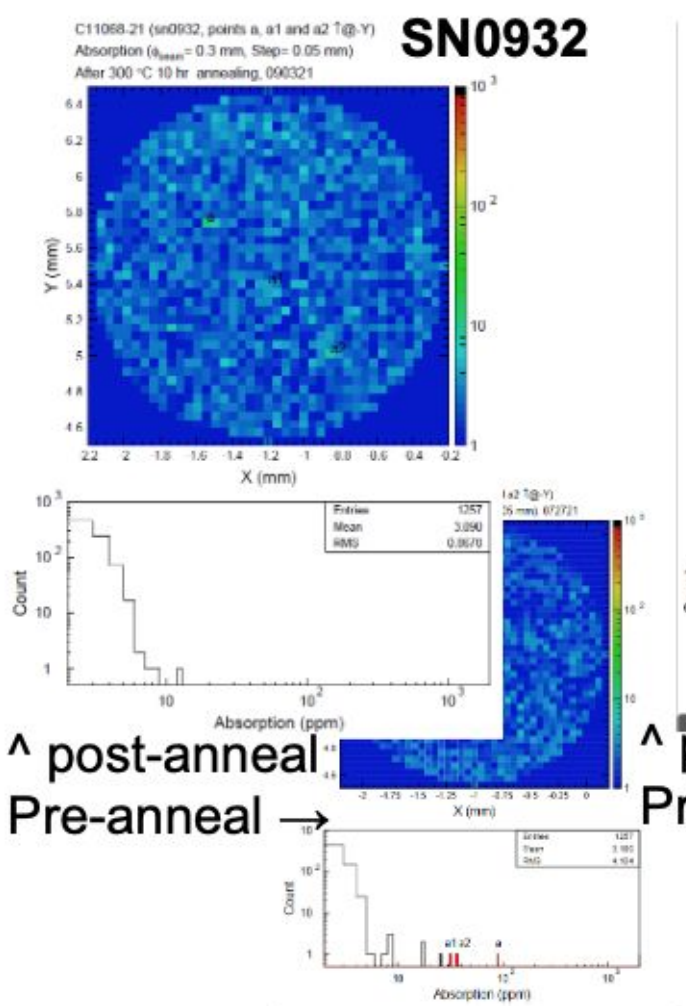
See small bump about 0.3-0.4nm tall at r=1mm.

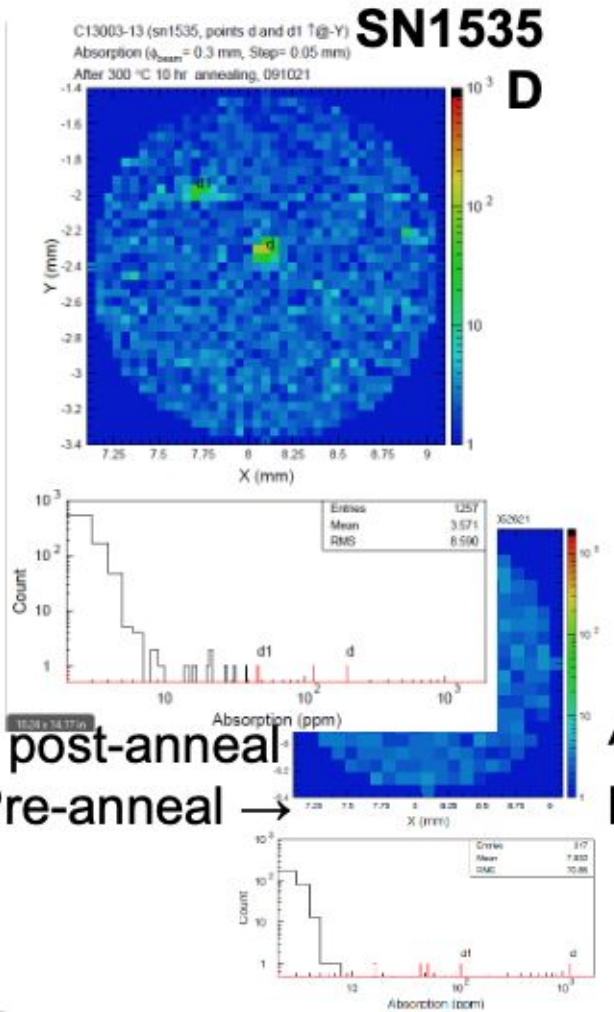
Corresponds to about 1.2ppm absorption. (600-1000ppm in RTS)

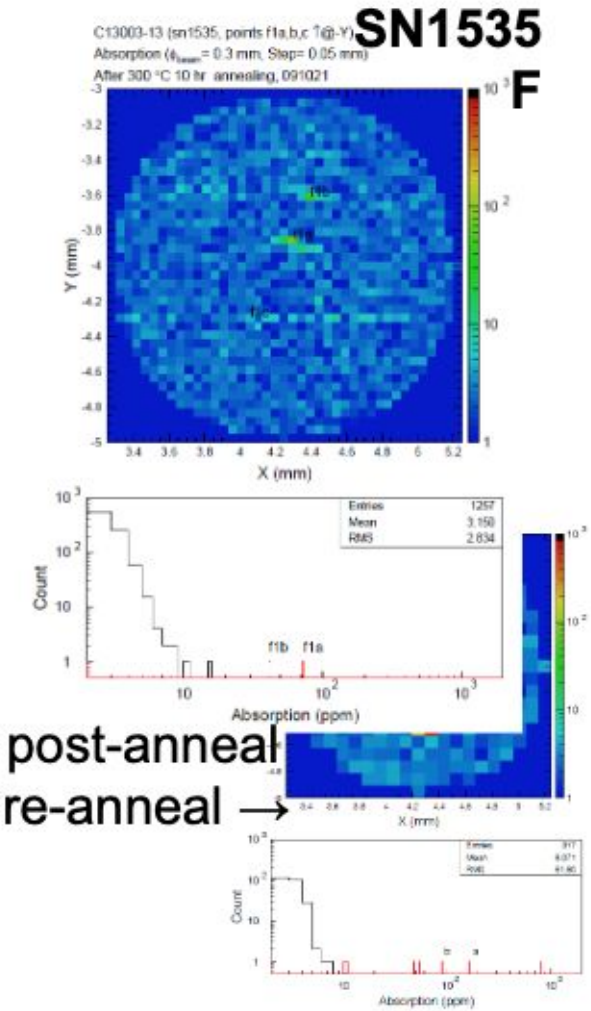
Annealed

- Run Parameters: 300 °C for 10 hours with ramp of 100 °C.

- SN0932
  - Ref. E2000107
  - Pre: 50-100 ppm
  - Post: below noise floor
- SN1535
  - Ref. E2000023
  - Ref. T2100216
  - ~5.9x drop at d
  - ~3.6x drop at f1a and f1b







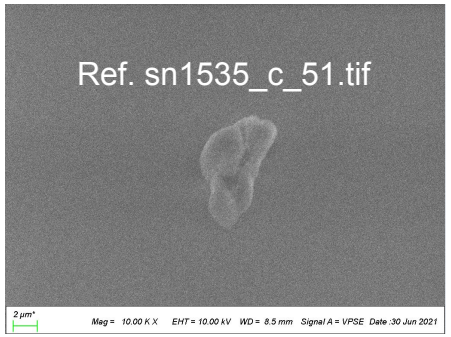
SN1535 after plasma clean and annealing

Original spot d “od” is now 2 spots: od1, od2

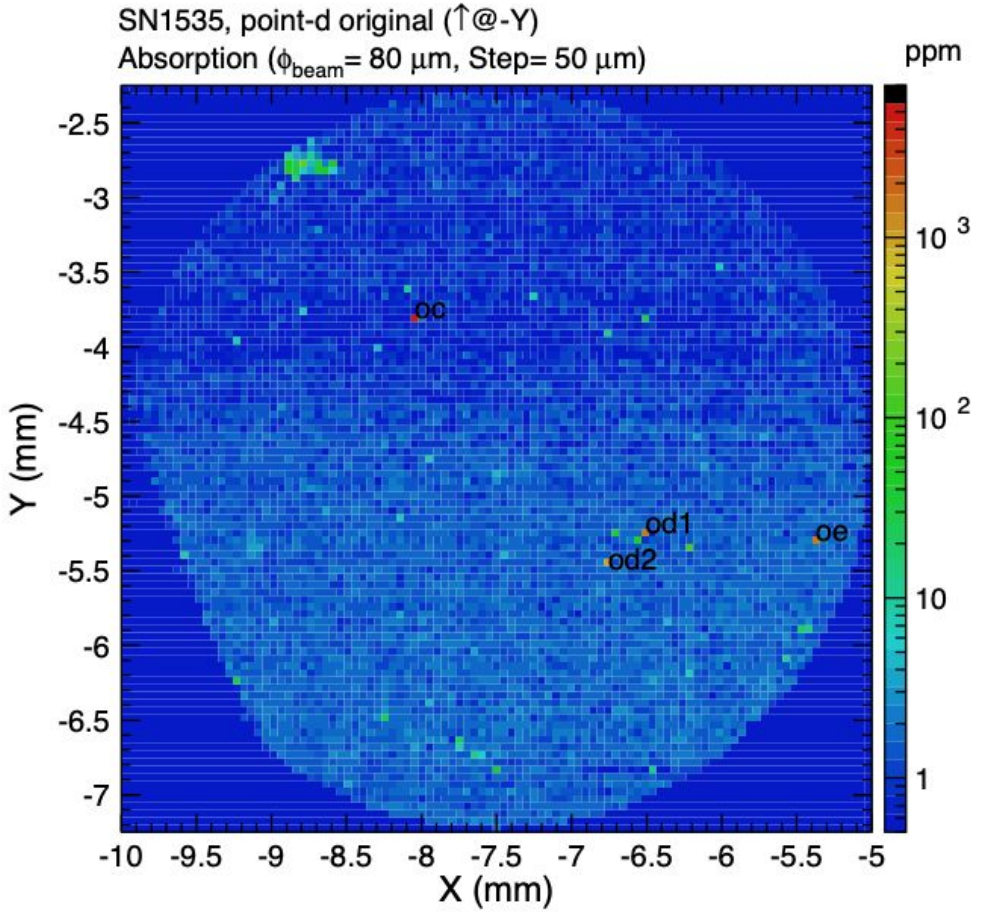
prior test resolution used 300 µm beam and step.

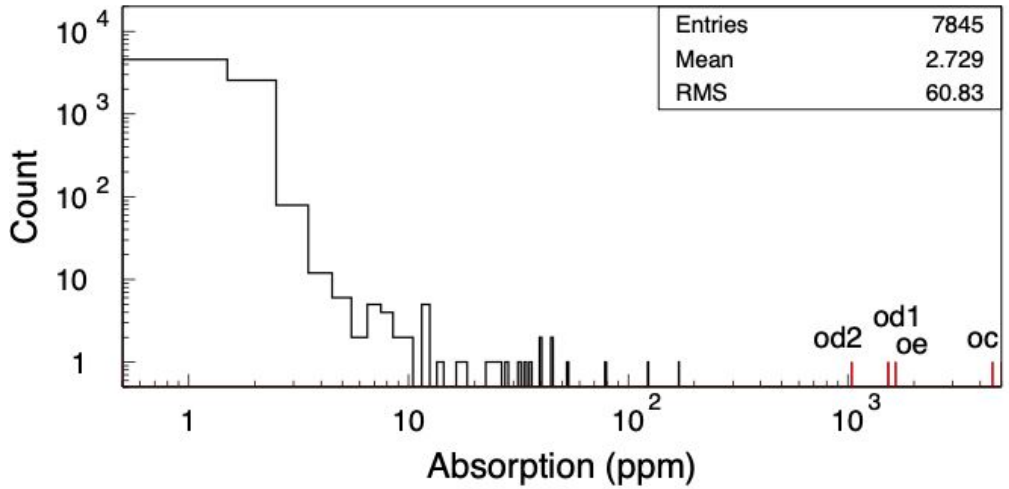
See DCC for more images

No	X_{optic} (mm)	Y_{optic} (mm)	Absorption (ppm)	Comment /Original ID
1	-8.014	-3.787	4546	oc?
2	-6.547	-5.239	1582	od1?
3	-5.345	-5.288	1648	oe?
4	-6.768	-5.439	1039	od2?
M1	11.067	-0.807		
M2	0.591	-12.108		next to ^
M3	-11.326	-2.102		

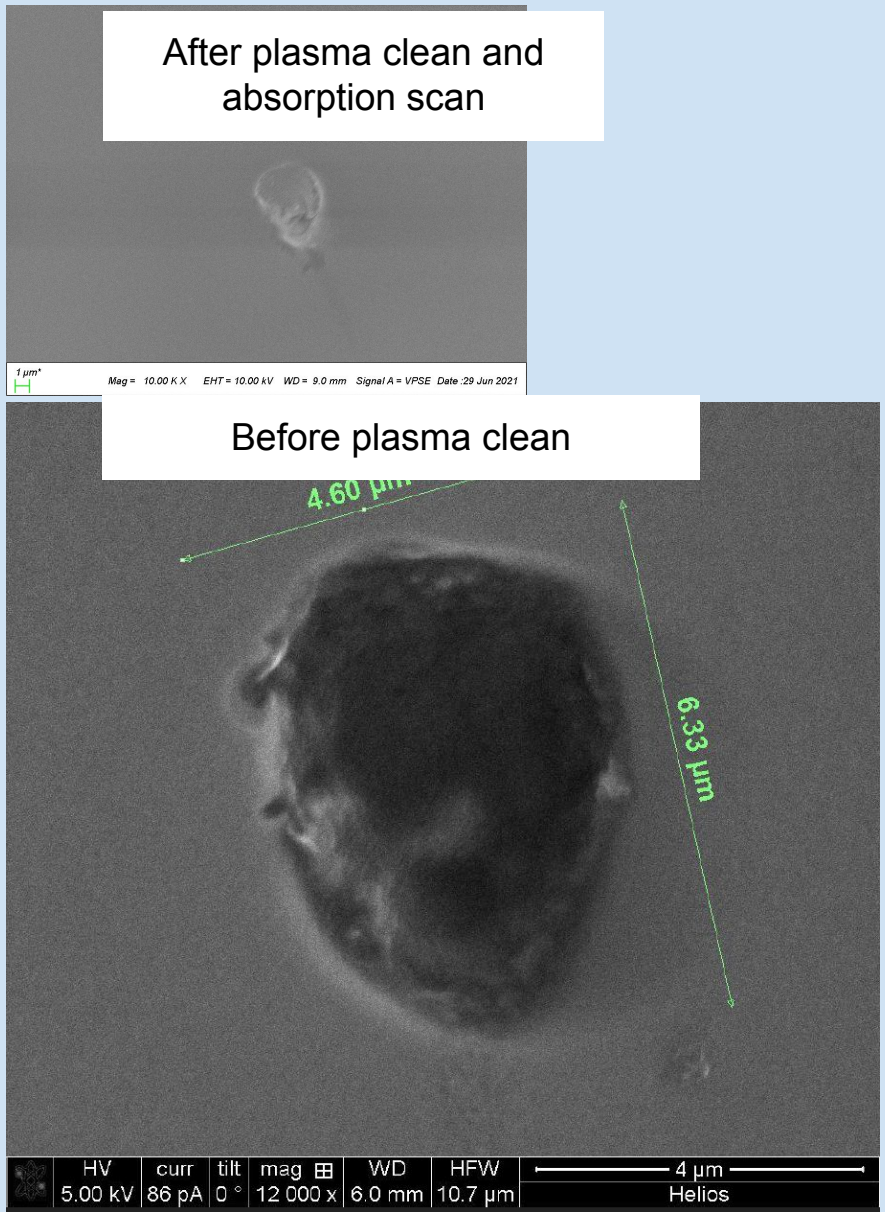


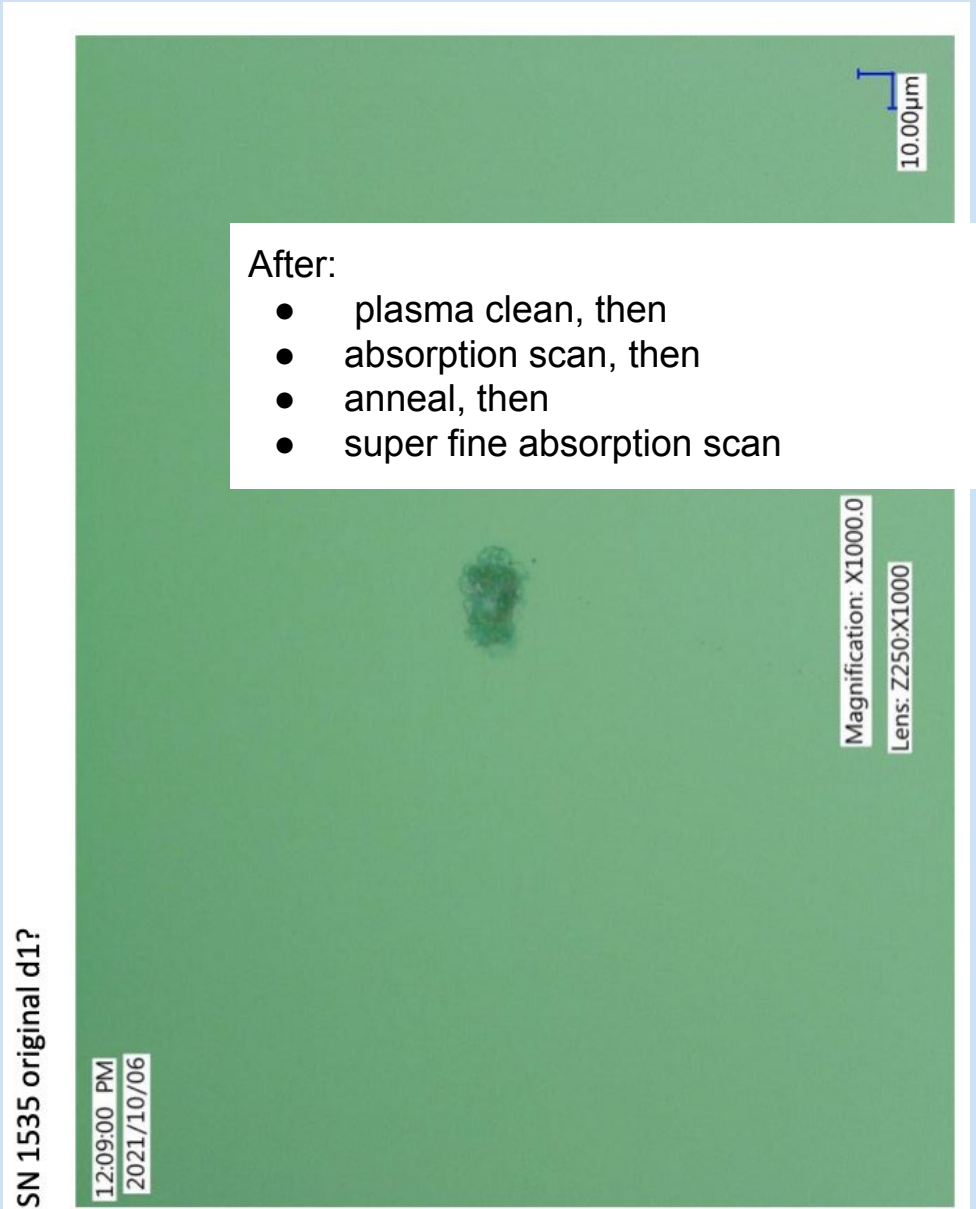
SN 1535 original d1?





od after plasma clean then annealing (green background) from most recent RTS scan does not compare well to SEM images?



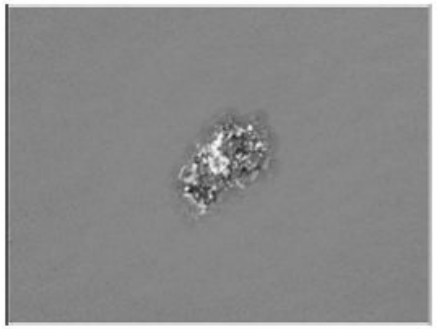


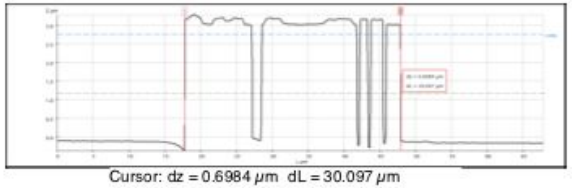
Ablation of SN1535 at PhotoMachining, 5 Nov 2021

Before Ablation, feature ‘od’

Topography 1360 x 1024 data points
Area: 116.87 x 87.98 µm²
Fri Nov 05 10:28:43 2021

Title: SN1535 Befre Ablation D
Comment: (None)
File: Z:\Cu...35 Befre Ablation D.plu
Sample ID:





Cursor: dz = 0.6984 µm dL = 30.097 µm

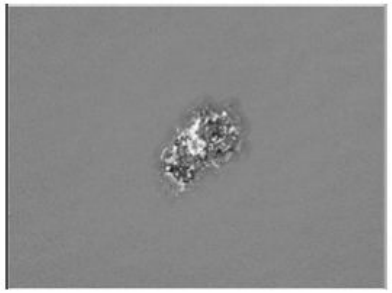
Display:	Profile	Term 3D:	None
Objective:	EPI 150X-L	Term 2D:	None
Z Scan:	20.0 µm	Filter 3D:	None
Z:	Non Inverted	Filter 2D:	None
Scale:	pv		
Zoom:	1X		

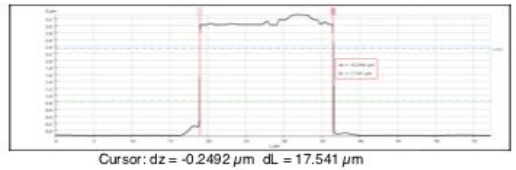
3.27 µm
-0.37 µm

Profile Parameters	Height:		Spacing:	
	Pa:	1.561 µm	Sm:	5.642 µm
	Pq:	1.593 µm	Pc:	73.69 peaks/mm
	Pp:	2.107 µm	Shape:	
	Pv:	1.541 µm	Psk:	2.971 µm
	Pt:	3.648 µm	Pku:	5.780 µm
	Ppm:	? µm	Hybrid:	
			Dela:	0.608 mrad
			Delq:	3.170 mrad

Topography 1360 x 1024 data points
Area: 116.87 x 87.98 µm²
Fri Nov 05 10:28:43 2021

Title: SN1535 Befre Ablation D
Comment: (None)
File: Z:\Cu...35 Befre Ablation D.plu
Sample ID:





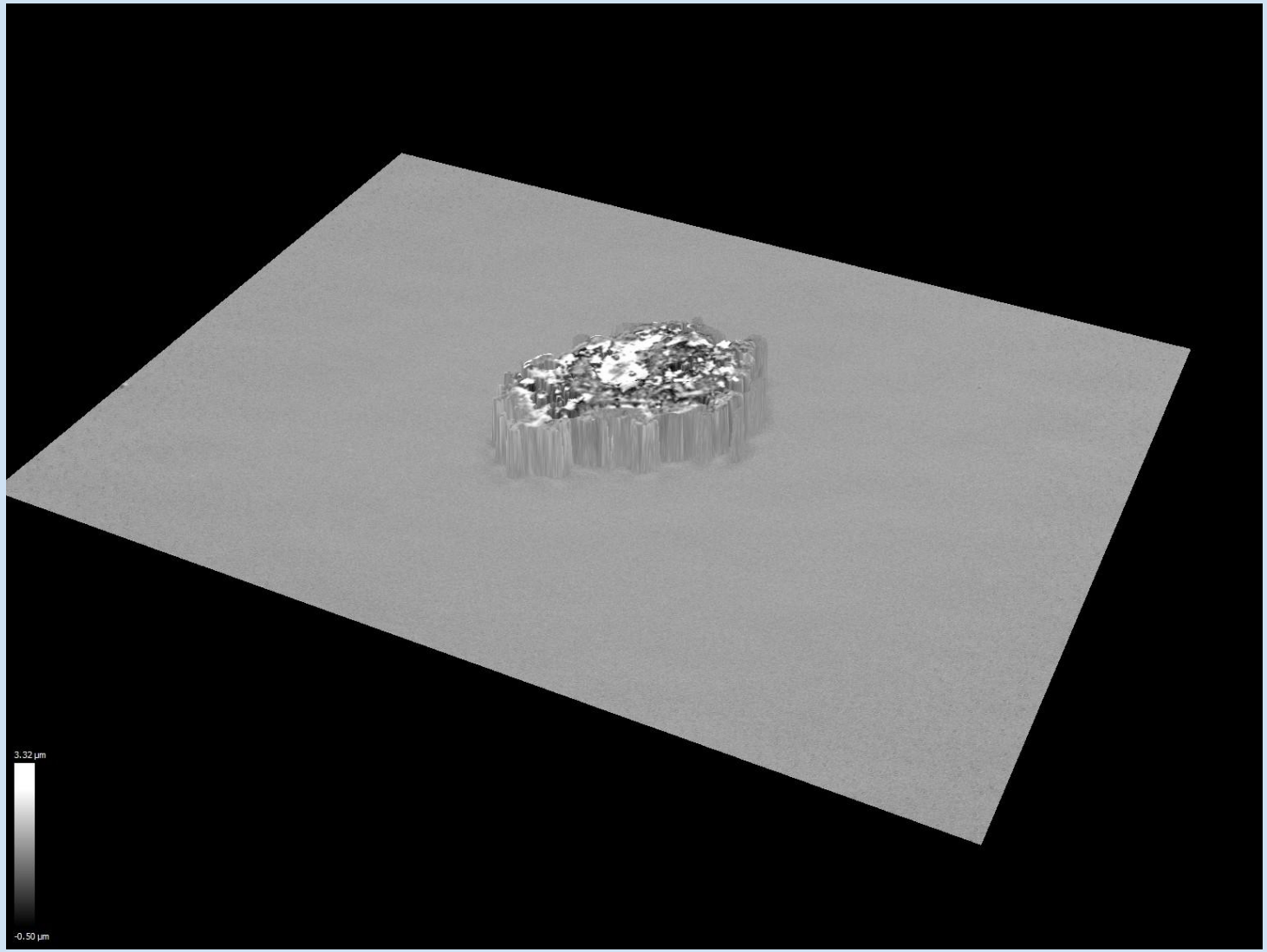
Cursor: dz = -0.2492 µm dL = 17.541 µm

Display:	Profile	Term 3D:	None
Objective:	EPI 150X-L	Term 2D:	None
Z Scan:	20.0 µm	Filter 3D:	None
Z:	Non Inverted	Filter 2D:	None
Scale:	pv		
Zoom:	1X		

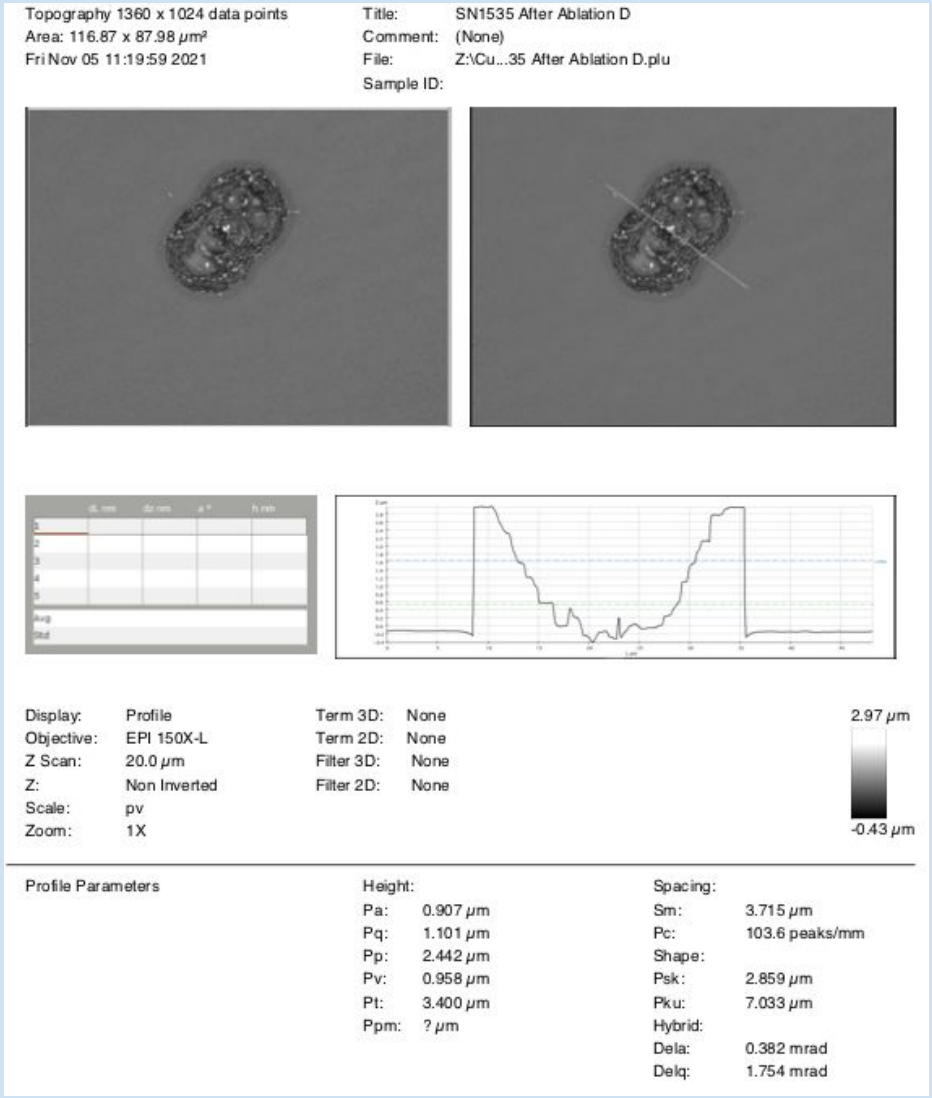
3.30 µm
-0.17 µm

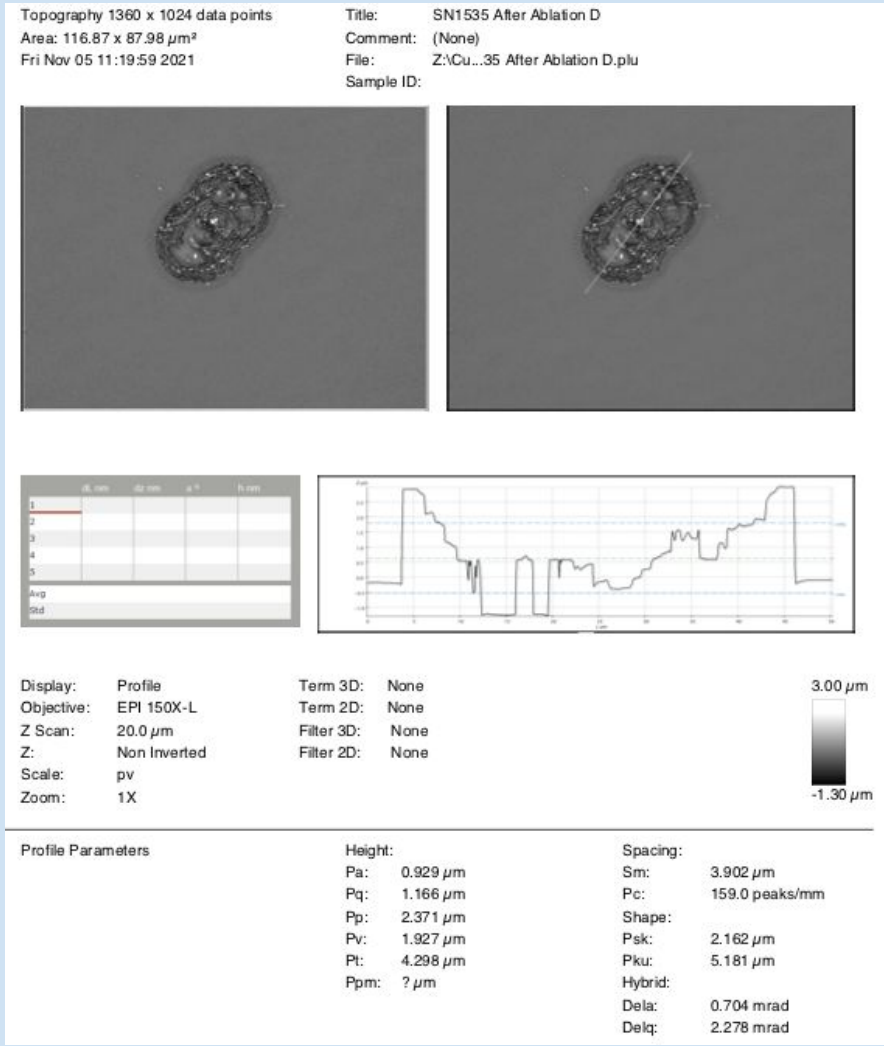
Profile Parameters	Height:		Spacing:	
	Pa:	1.377 µm	Sm:	19.09 µm
	Pq:	1.493 µm	Pc:	17.46 peaks/mm
	Pp:	2.453 µm	Shape:	
	Pv:	1.018 µm	Psk:	2.721 µm
	Pt:	3.471 µm	Pku:	5.644 µm
	Ppm:	? µm	Hybrid:	
			Dela:	0.165 mrad
			Delq:	1.530 mrad

SN1535, od, before ablation



SN1535, od, after ablation: 8 pulses, 7 uJ each, in 2 partially overlapping spots





SN1535, ablation tests in clean coating

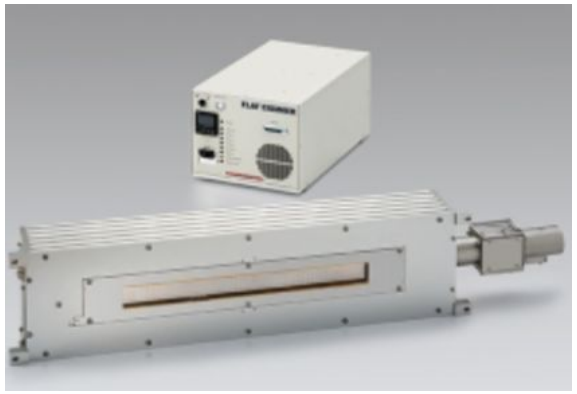
- First spot: 600 um in +X direction from ‘od’, 10 pulses
- Second spot: additional 300 um in +X direction, 2 pulses

Next Steps

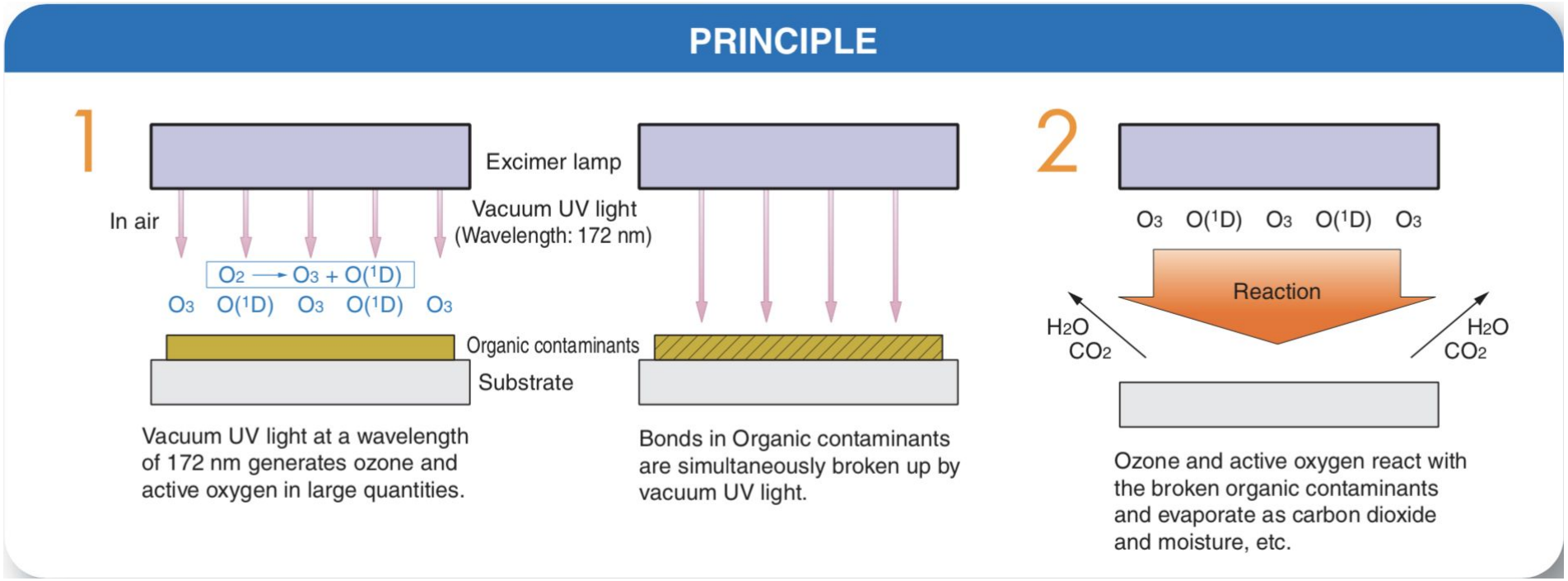
SEM at Caltech - ✔
SEM ✔
RTS ✔
HWS✔
Anneal✔
Confirm status of d(old)✔ Oct ‘21
Send to MIT for ablation of original spot d - aluminum✔ Nov, ‘21
RTS
Anneal/ or UV light
Chemical Etch?
Absorption

Extra Slides

Excimer Lamp: Carbon removal with UV-O_3

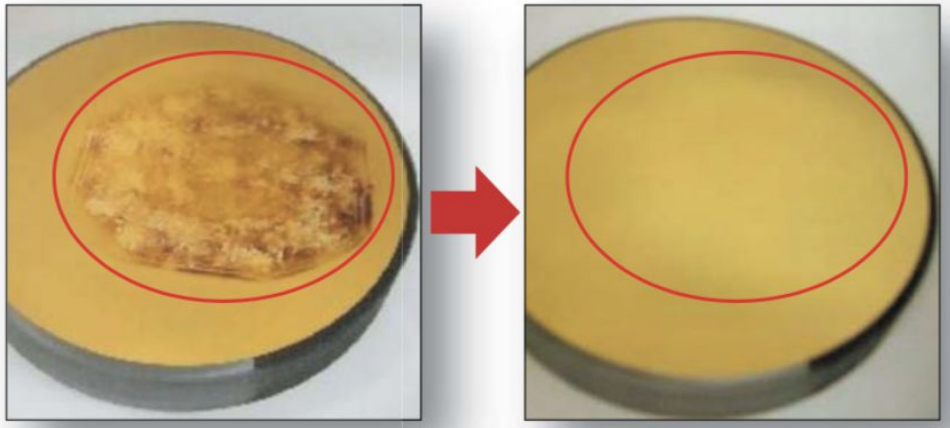


Vacuum UV light at a wavelength of 172 nm emitted from an excimer lamp is greatly absorbed by oxygen so that highly concentrated active oxygen can be generated. Vacuum UV light is also capable of breaking the molecular bonds of organic matter and so provides benefits in various processes such as accelerating the cleaning speed, improving the cleaning quality.



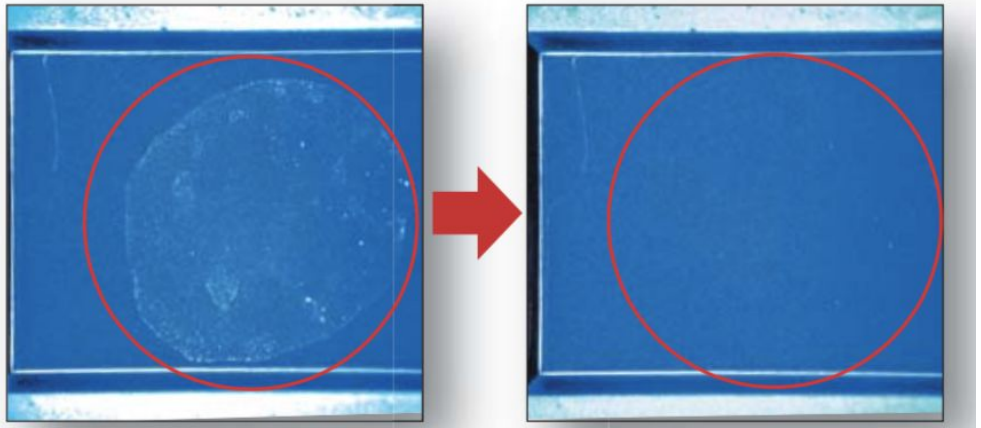
Excimer Lamp: Carbon removal with UV-O_3

■Optical cleaning of evaporated gold coatings on laser mirrors



Before excimer light irradiation After excimer light irradiation

■Removal of acetone cleaning residues



Before excimer light irradiation After excimer light irradiation

2-4. Contaminants removable by UV-O_3 cleaning

1. Cutting oils
2. Mixtures of beeswax and pine resin
3. Lapping agents
4. Vacuum-pump oils
5. Silicon diffusion-pump oils
6. Silicon vacuum greases
7. Soldering fluxes
8. Human sebum
9. Contaminants adsorbed during long-term air exposure
10. Carbon thin films formed by vacuum evaporation

Bond	Bond energy (KJ/mol)	Bond	Bond energy (KJ/mol)
O - O	138.9	C = C	607
O = O	490.4	C ≡ C	828
O - H	462.8	C = O	724
C - C	347.7	C - Cl	328.4
C - H	413.4	H - F	563.2
C - N	291.6	C - F	441.0
C ≡ N	791	H - Cl	431.8
C - O	351.5	N - H	309.8

Table 3. Chemical-bond energy [2)]

E(172nm) = 692 kJ/mol

Ta - O 839 kJ/mol
Si - O 799 kJ/mol

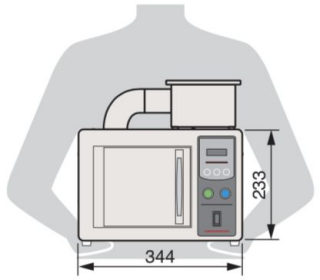
Excimer Lamp: Carbon removal with UV-O_3

FLAT EXCIMER™ EX-mini

■Irradiation area size
86 mm × 40 mm

R&D Compact

Amazingly handy and easy to use design allows simple yet highly accurate testing and evaluation in any place needed.



Weight: 6.5 kg
(Unit: mm)

SPECIFICATIONS

Parameter		Description / Value
Emission wavelength		172 nm
Irradiance ①	Typ.	65 mW/cm^2
Irradiation area size (W×H)		86 mm × 40 mm
Lamp design life ②		2000 h
Input voltage (AC)		100 V to 240 V
Power consumption		150 VA or less
Cooling method		Forced air cooling by fan or duct
Duct suction air flow rate ③④		0.25 m^3/min to 0.35 m^3/min
Operating / storage temperature range		+5 °C to +35 °C / -25 °C to +55 °C
Operating / storage humidity range		10 % to 80 % / below 80 % (no condensation)
Control method		Panel control / external control
Weight		6.5 kg

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Valid Until	Sales Engineer	Terms	Ship Via
5/6/2021	Mesa, Eric	Net 30 Days	Best Method

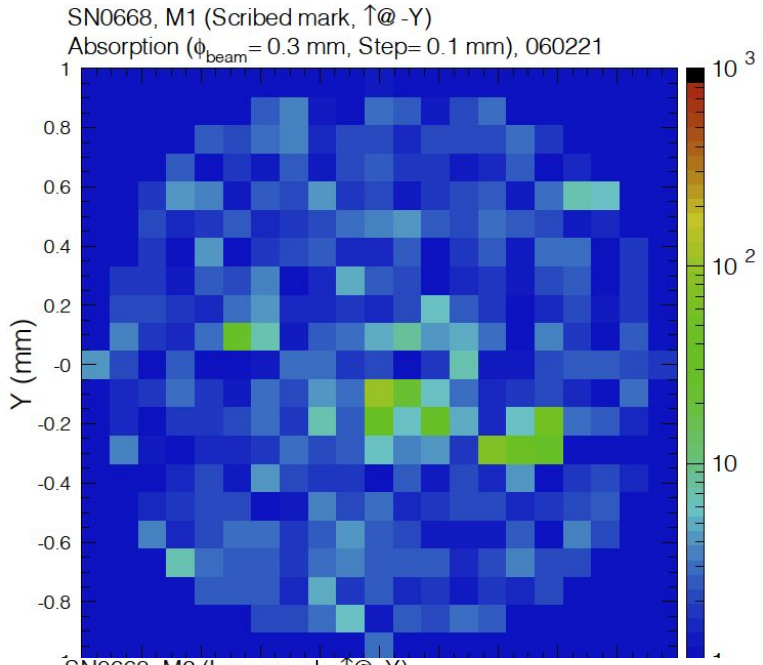
Product Number	Description	RoHS	Quantity	Price per Unit	Lead Time
L12530-01	Excimer Lamp Light Source	NA	1	$13258.00	*TBD

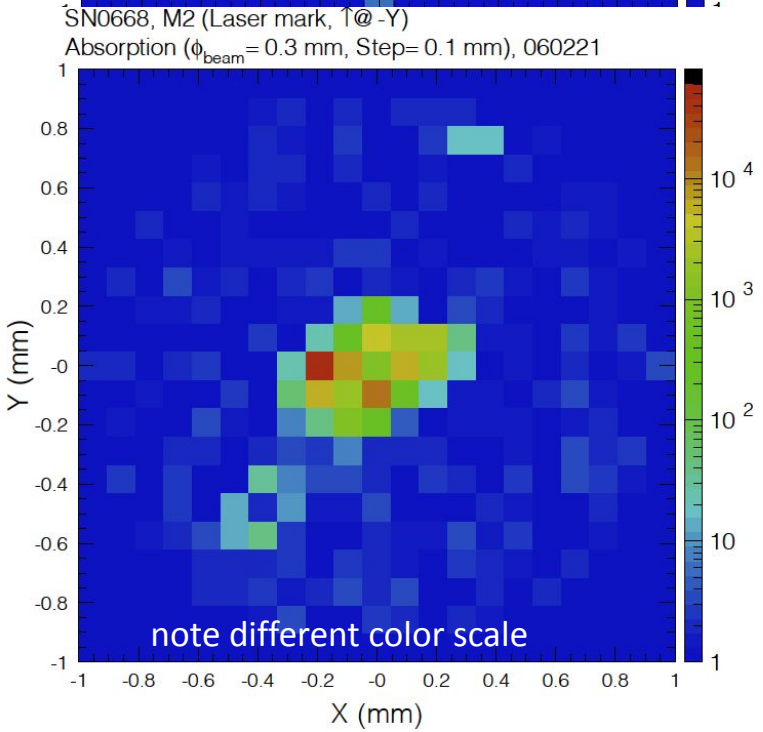
*Lead-time vary based on stock, production and materials availability. Lead-time is confirmed once an order is received and processed.

Notes on absorption signal

SN0668, M1, scribed mark

SN0668, M2, laser burned mark (on black ink)





Ink does absorb see T2000526