GENESIS Solar Wind Irradiation Collector Substrate Damage and Surface Contaminates

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Jacobs Technology

at NASA Johnson Space Center

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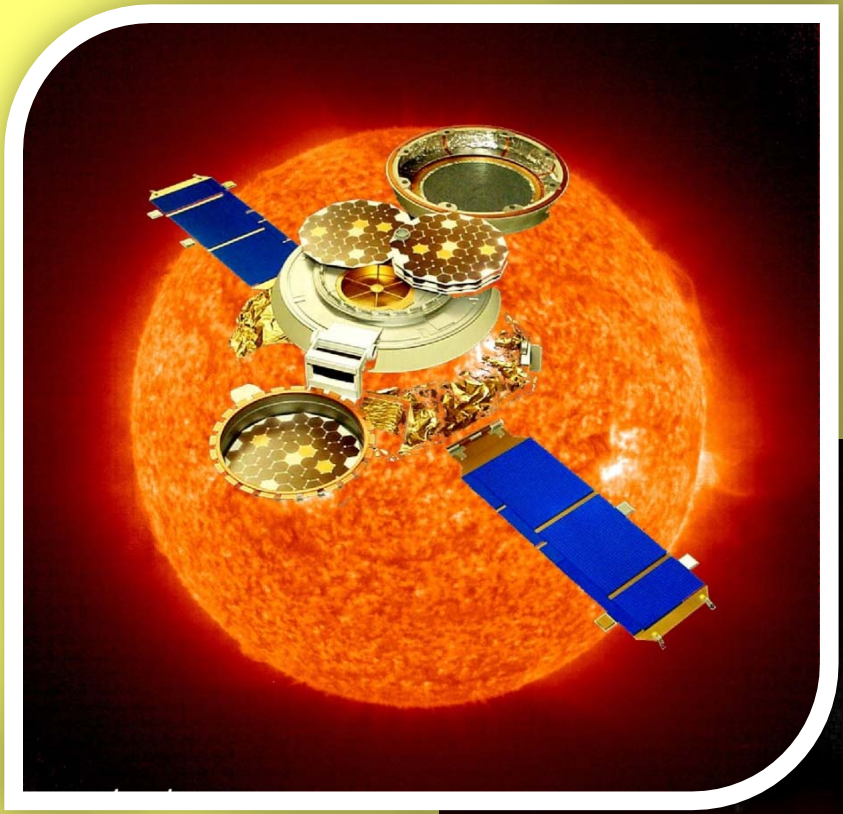
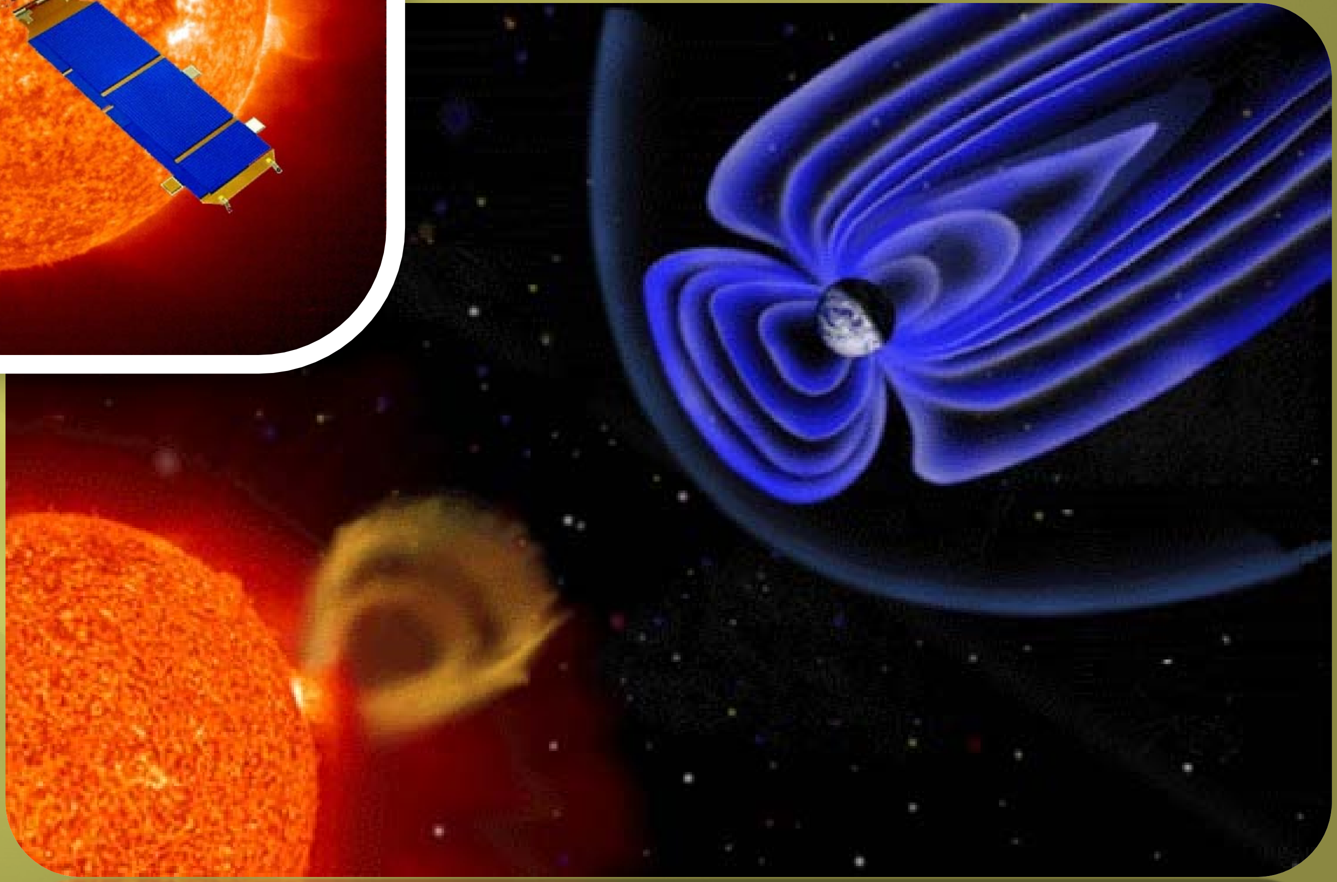
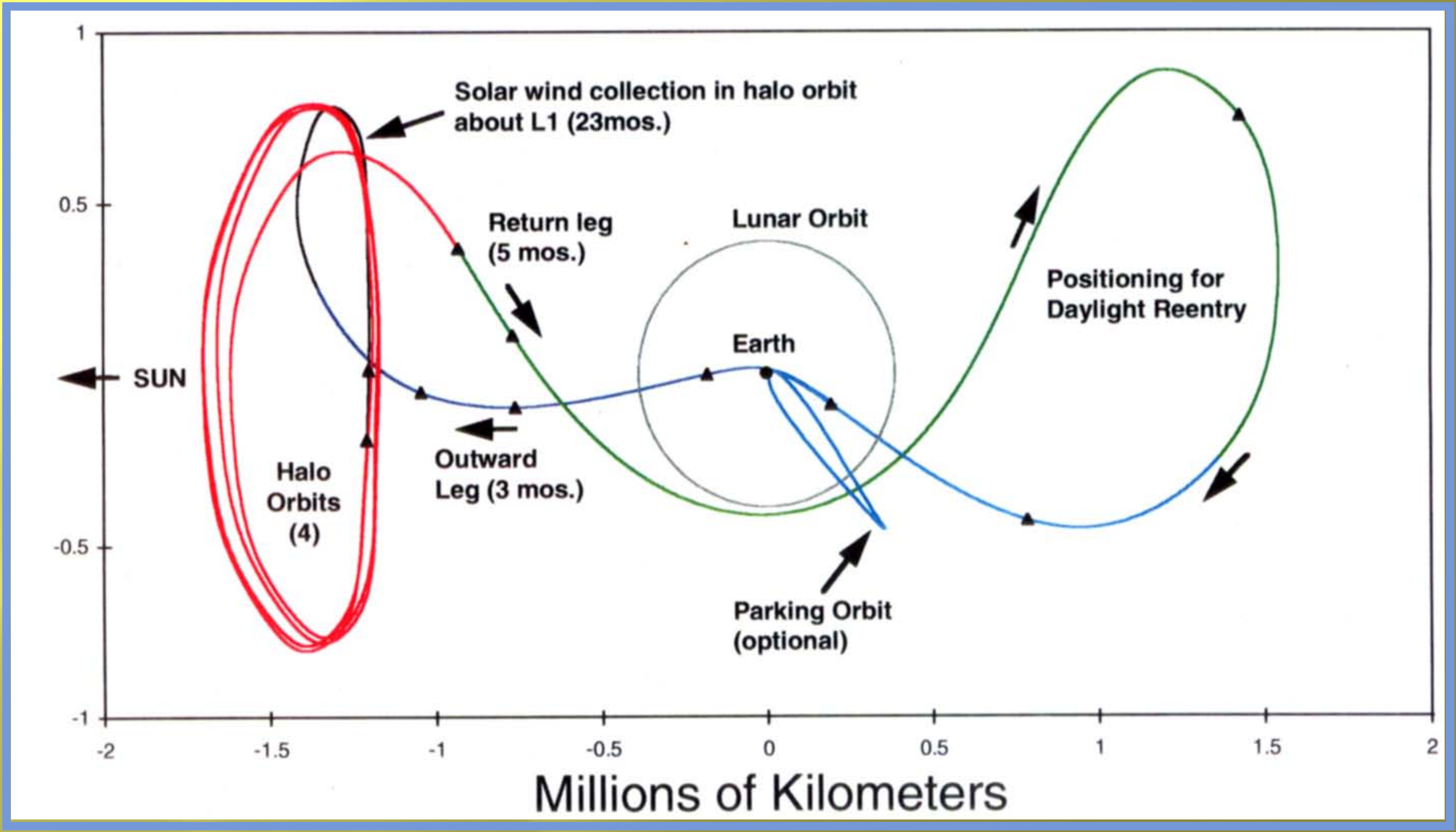
GENESIS Launch
Cape Canaveral
August 8, 2001

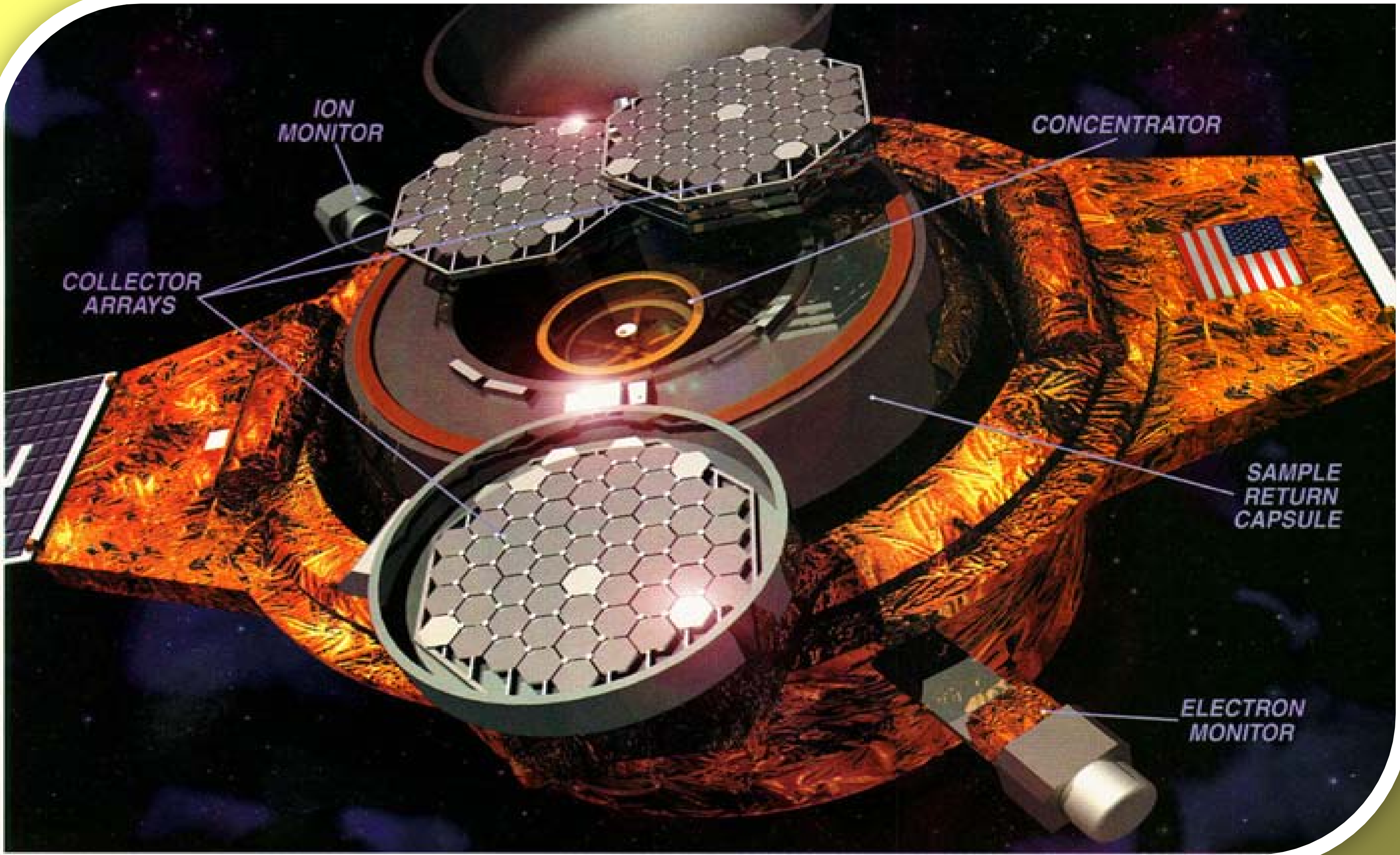


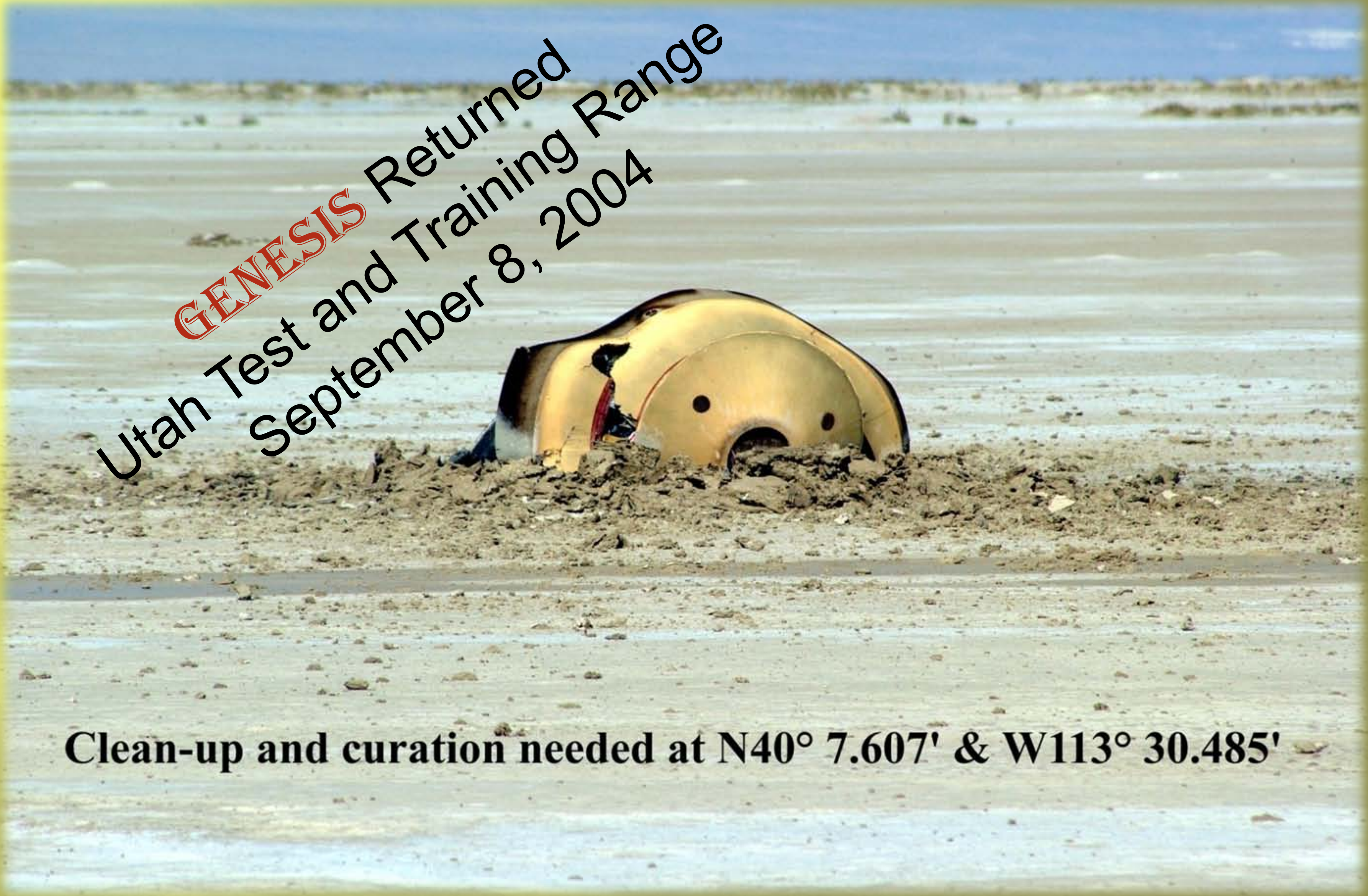
GENESIS needed to travel outside Earth’s magnetosphere



GENESIS Journeyed to Earth-Sun Lagrange 1

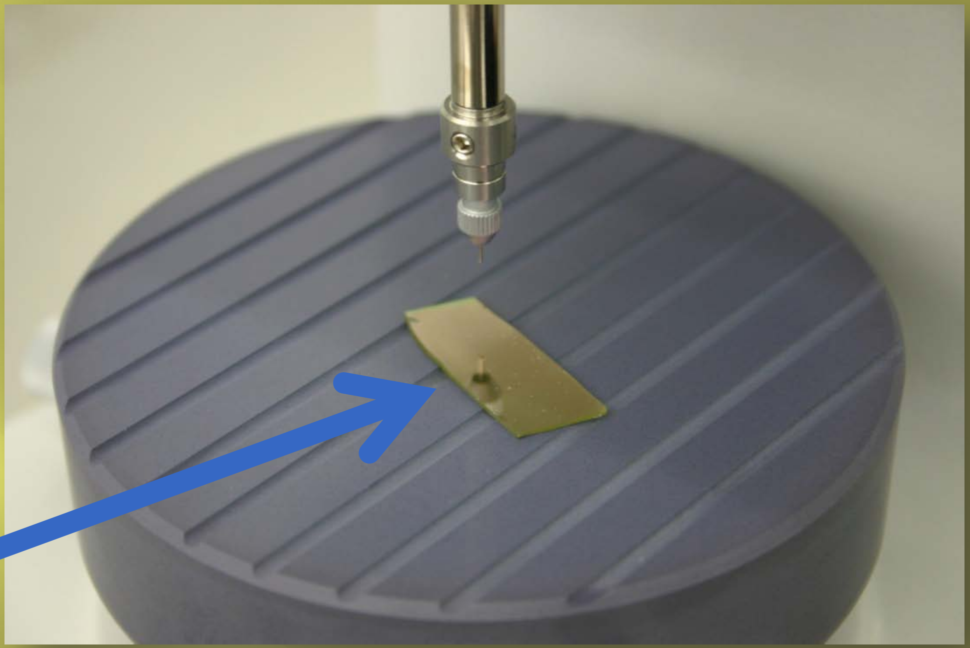



ION
MONITOR
CONCENTRATOR
COLLECTOR
ARRAYS
SAMPLE
RETURN
CAPSULE
ELECTRON
MONITOR


Genesis Returned
Utah Test and Training Range
September 8, 2004
Clean-up and curation needed at N40° 7.607' & W113° 30.485'


Landing Site
GENESIS
SEPTEMBER 8, 2004
2003 Landing Marker
~ 6 miles
1 mile

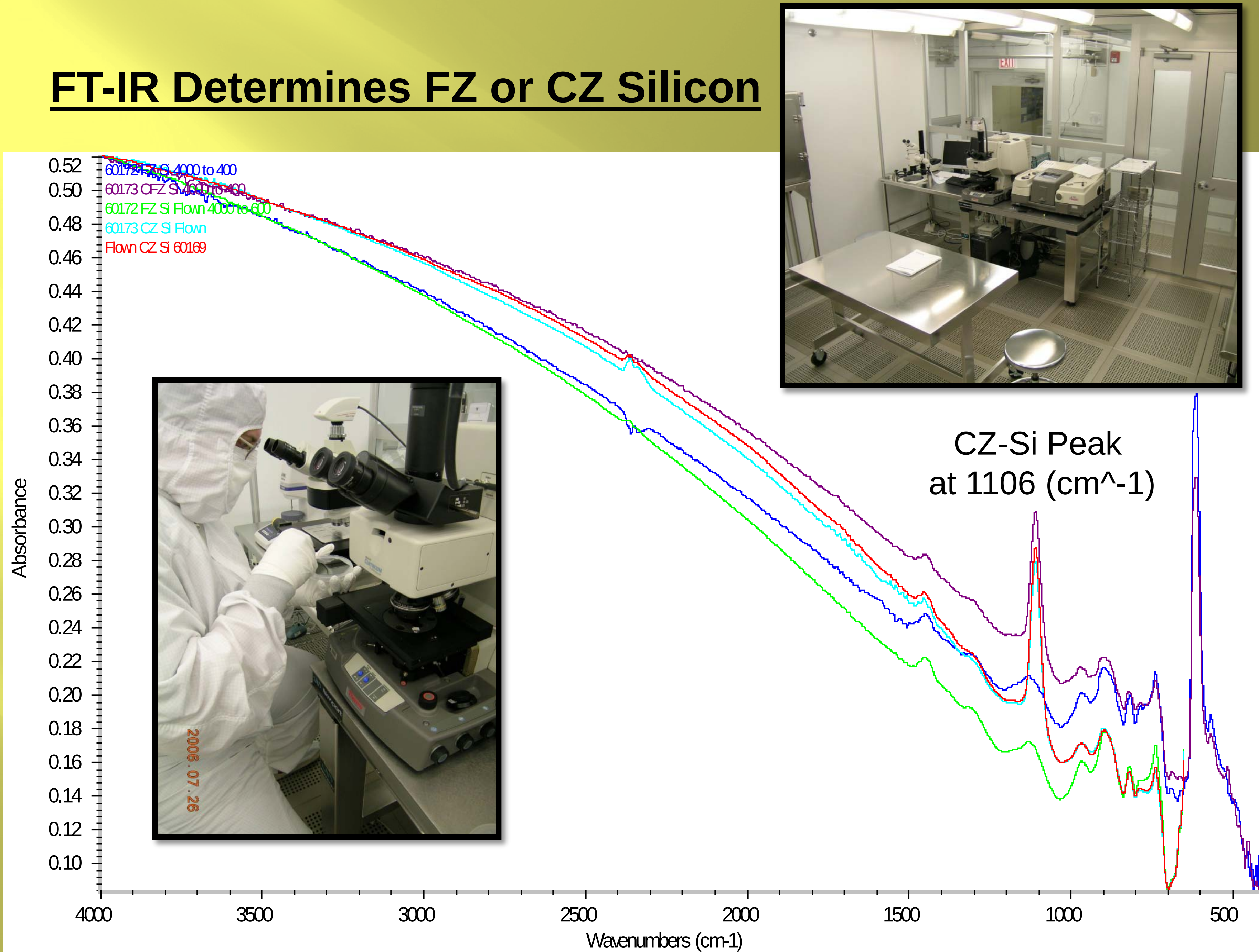
GENESIS Wafer Fragments



Wafer Thickness Measurement
Determines Solar Wind Regime

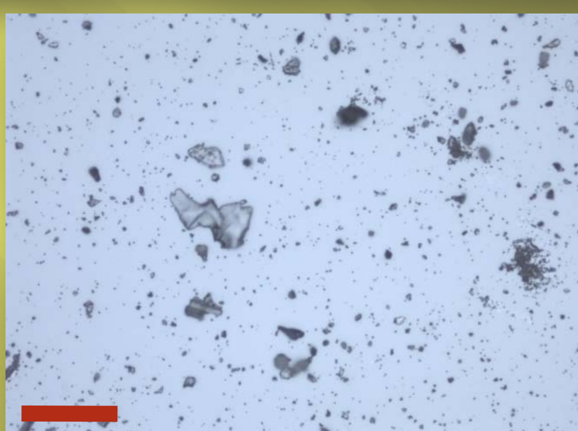
Bulk (B/C) collector arrays = ~700 µm
Coronal Mass Ejection (E) array = ~650 µm
High Speed (H) collectors = ~600 µm
Low Speed (L) collectors = ~550 µm

FT-IR Determines FZ or CZ Silicon



Microscopic Surface Characterization

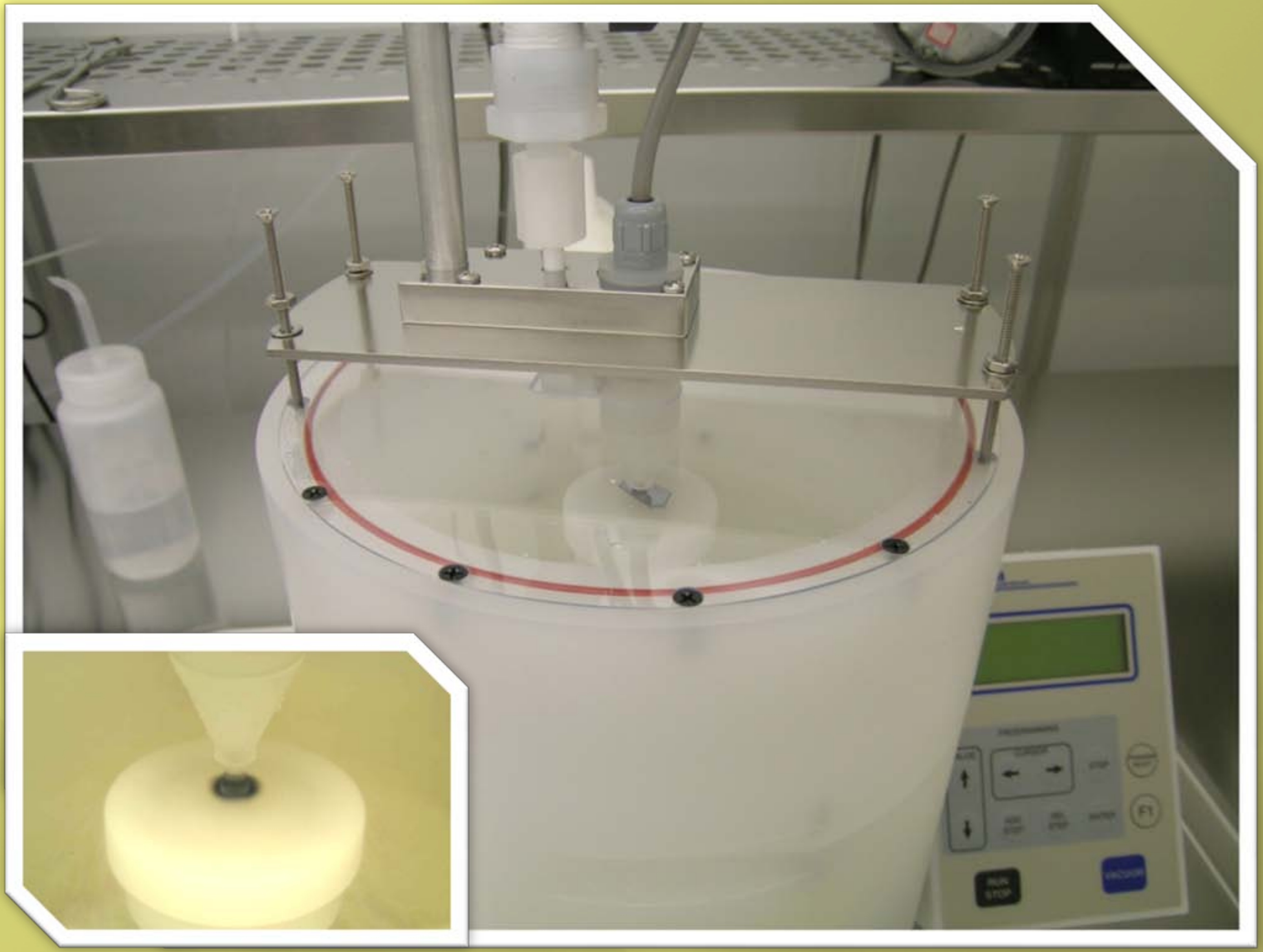
Leica DM6000 M
Automated Microscope



50 µm

GENESIS Ultra-Pure Water Megasonic Wafer Spin Cleaner

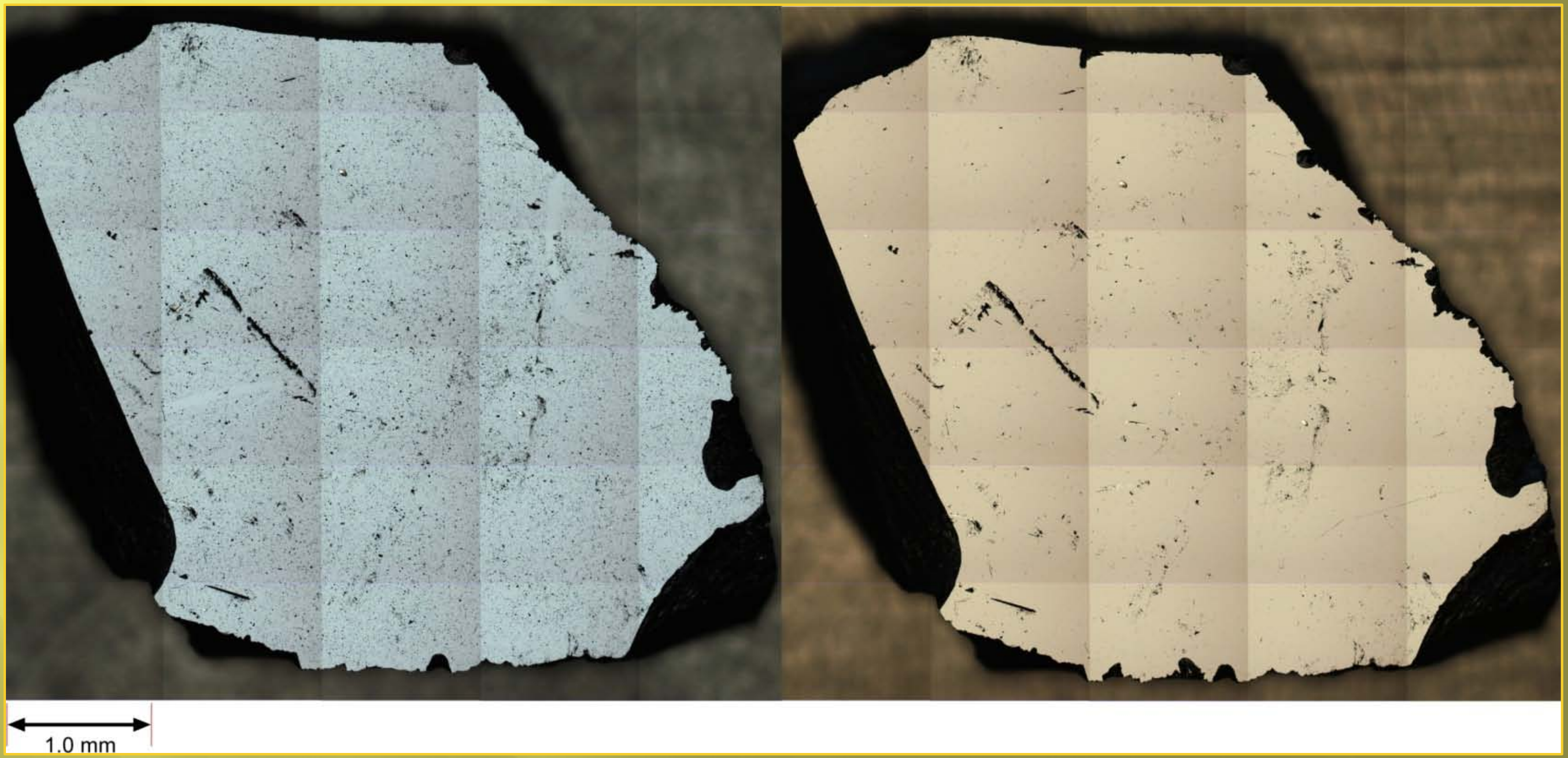
(NASA New Technology # 24499)



- 1.5 l/min , 40º C UPW flow
- 1 MHz, 0.4 A Sonication
- 3000 RPM Spin
- 24” Hg Vacuum Sample Holder
- 3 mm to 10 cm Genesis Samples

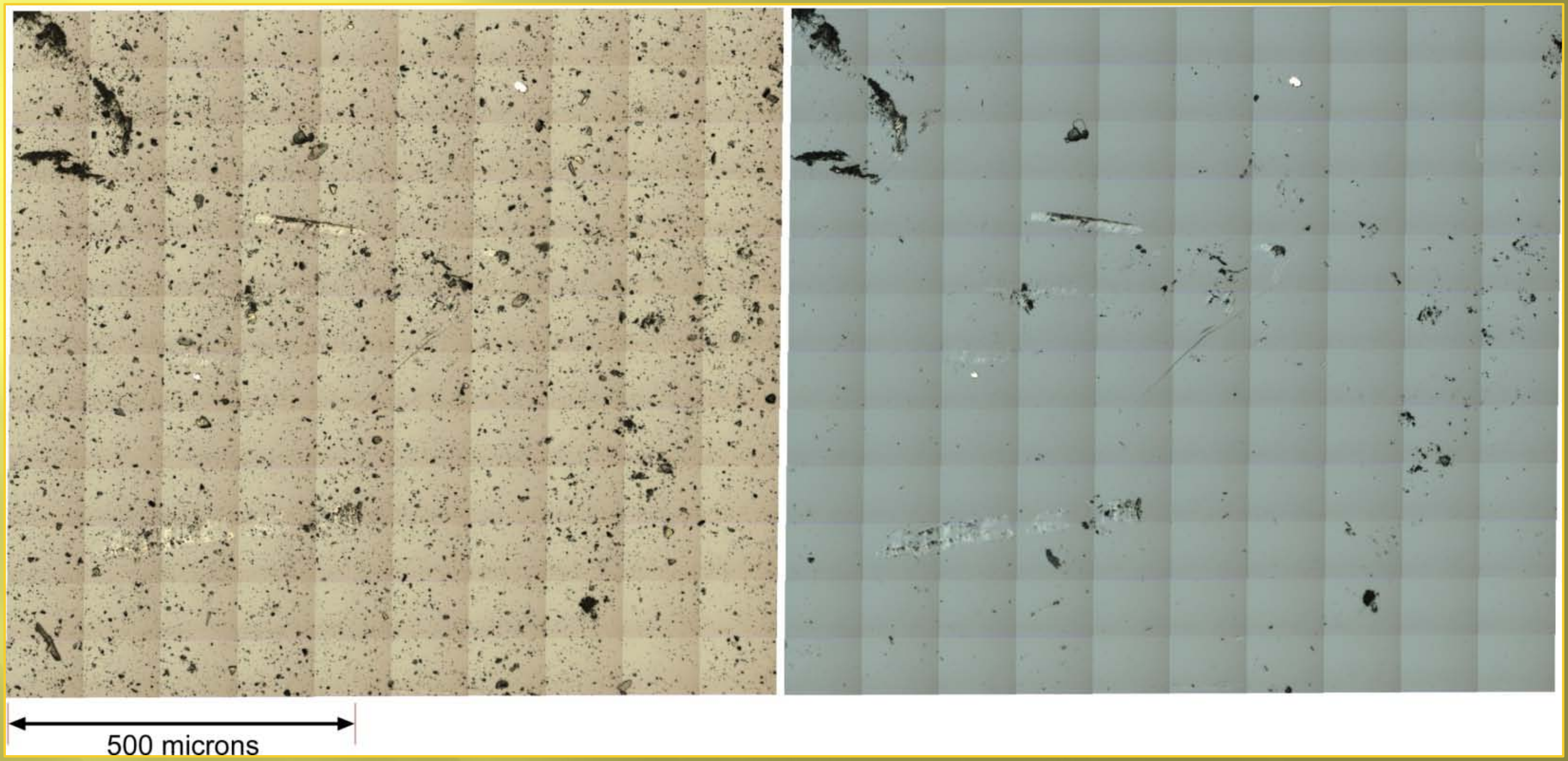
Genesis Si B/C array sample 60458

Before and After UPW/Megasonic Cleaning

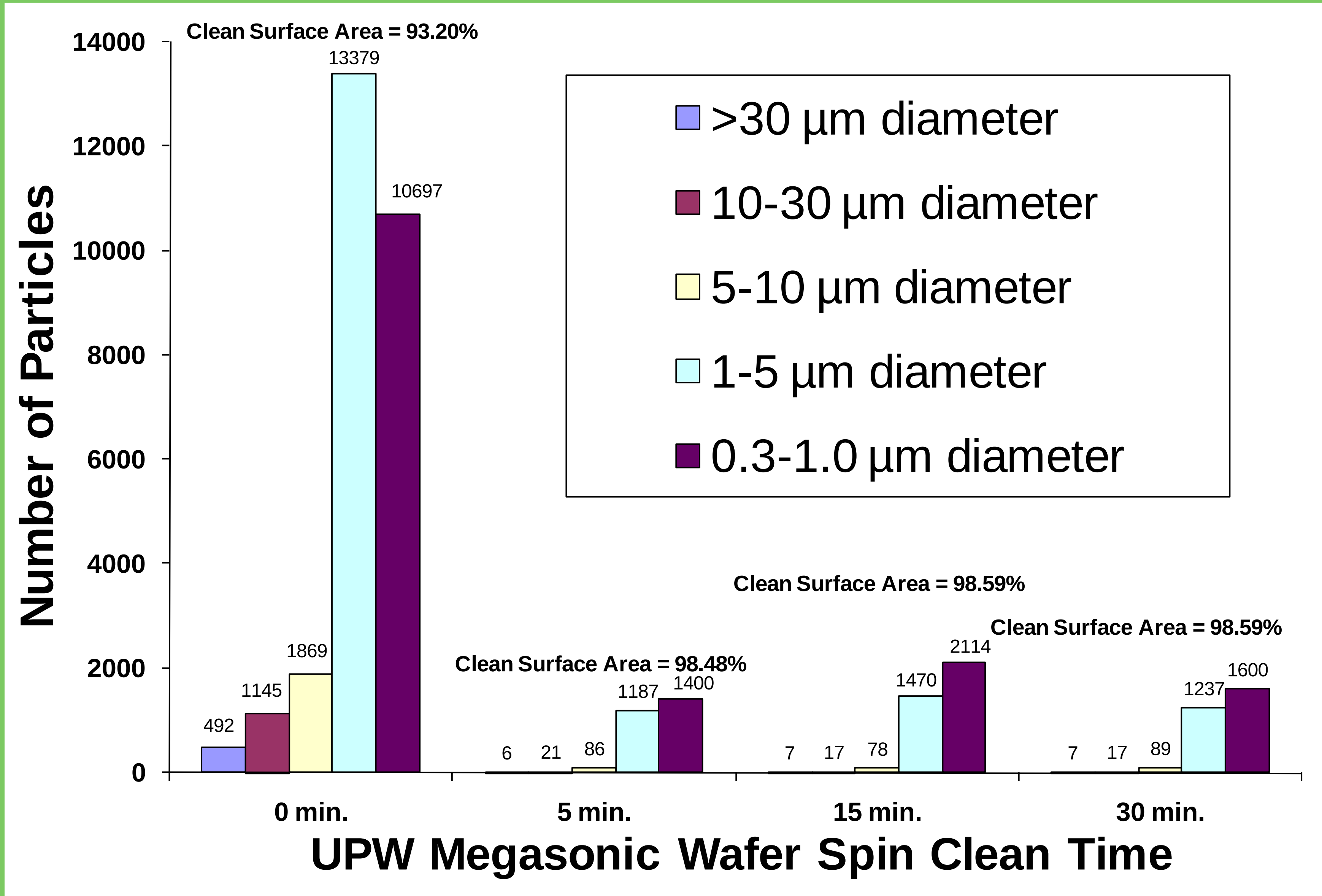


Genesis Si B/C array sample 60458

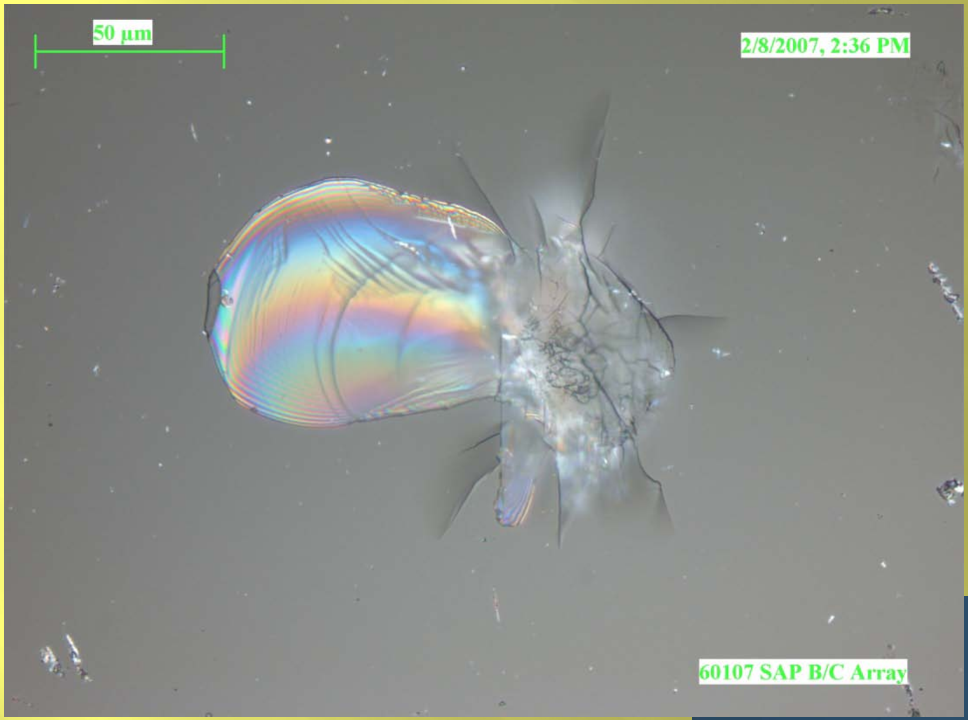
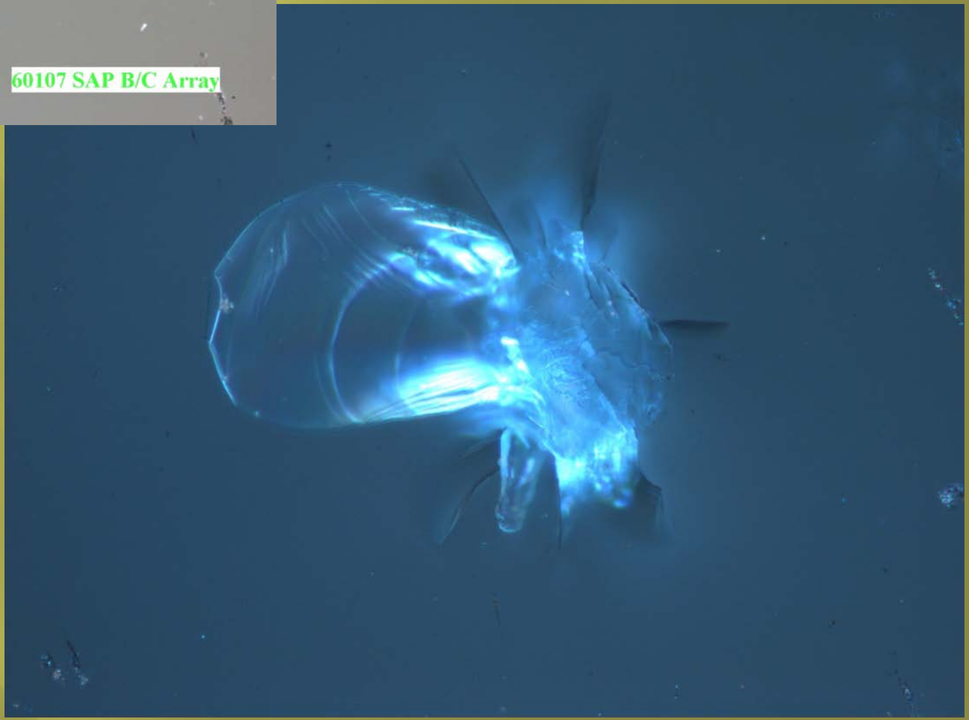
Before and After UPW/Megasonic Cleaning

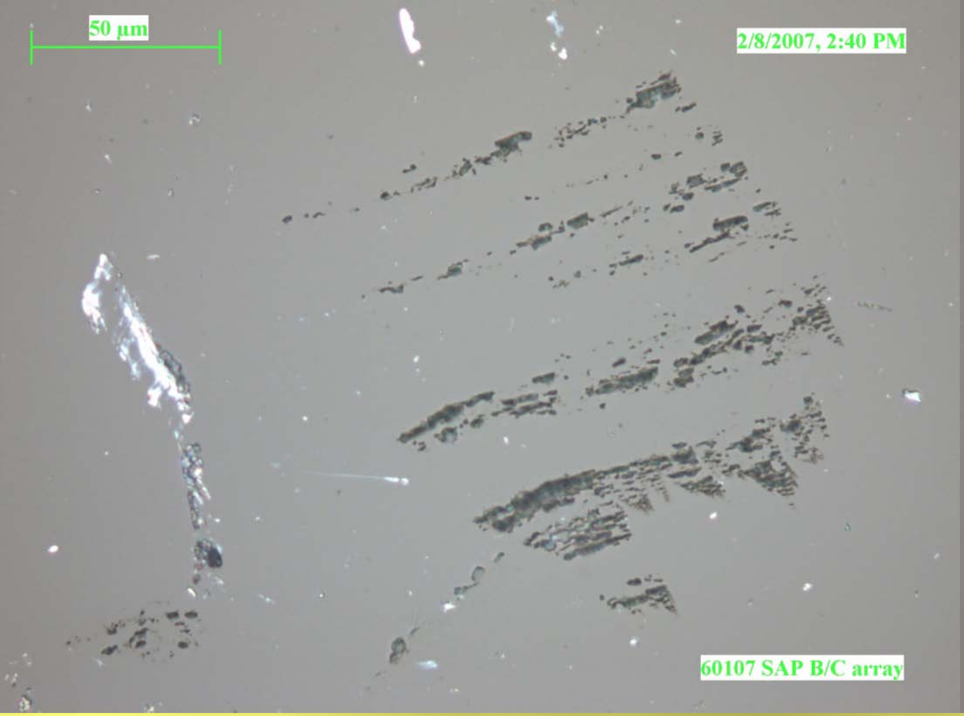


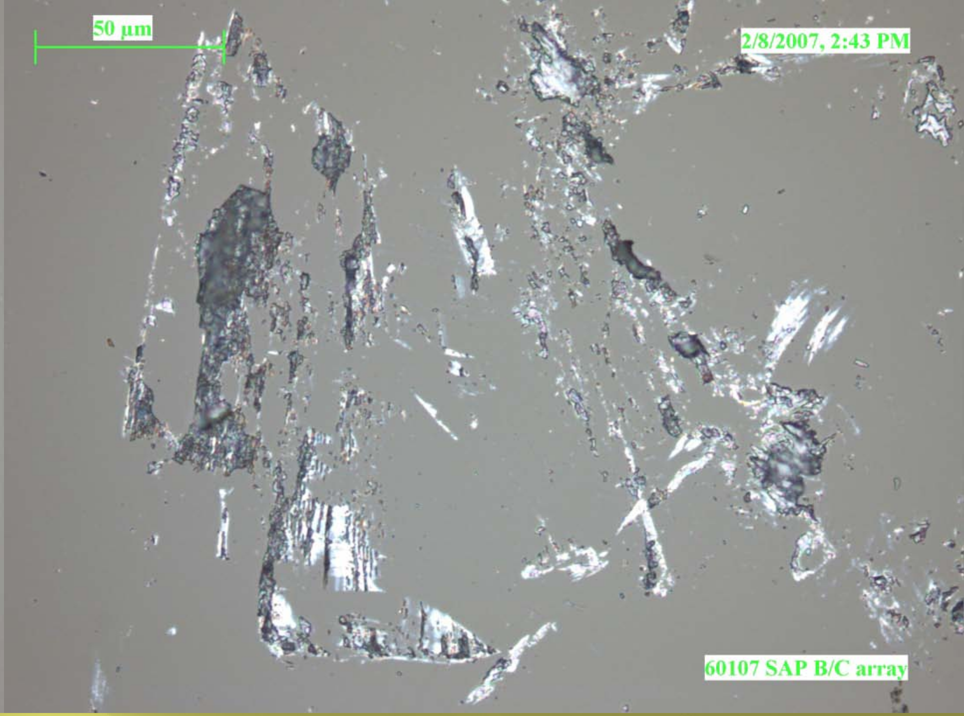
Taken with Leica DM 6000 M Optical Microscope with 50X lens

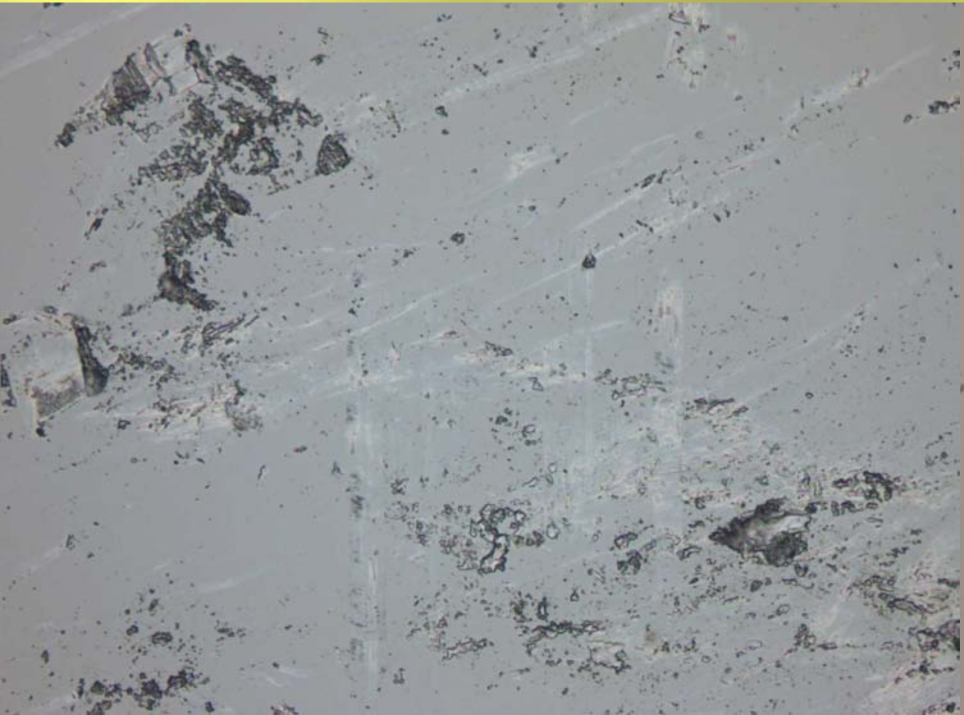


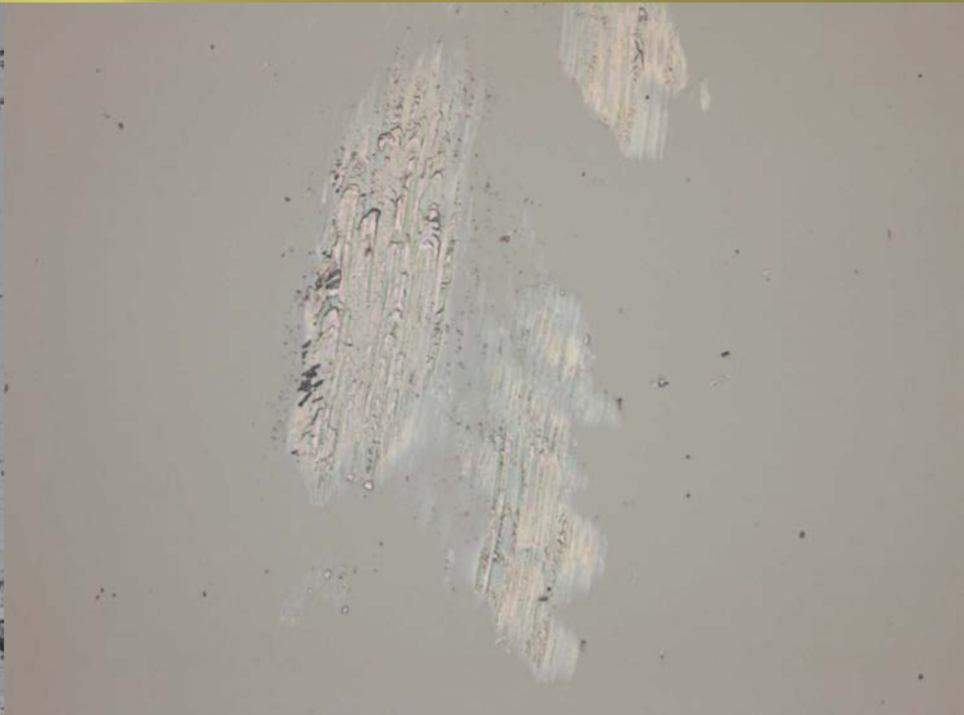
Number of Particles
14000
12000
10000
8000
6000
4000
2000
0
Clean Surface Area = 93.20%
13379
10697
1869
1145
492
Clean Surface Area = 98.48%
6
21
86
1187
1400
Clean Surface Area = 98.59%
7
17
78
1470
2114
Clean Surface Area = 98.59%
7
17
89
1237
1600
>30 µm diameter
10-30 µm diameter
5-10 µm diameter
1-5 µm diameter
0.3-1.0 µm diameter
0 min.
5 min.
15 min.
30 min.
UPW Megasonic Wafer Spin Clean Time

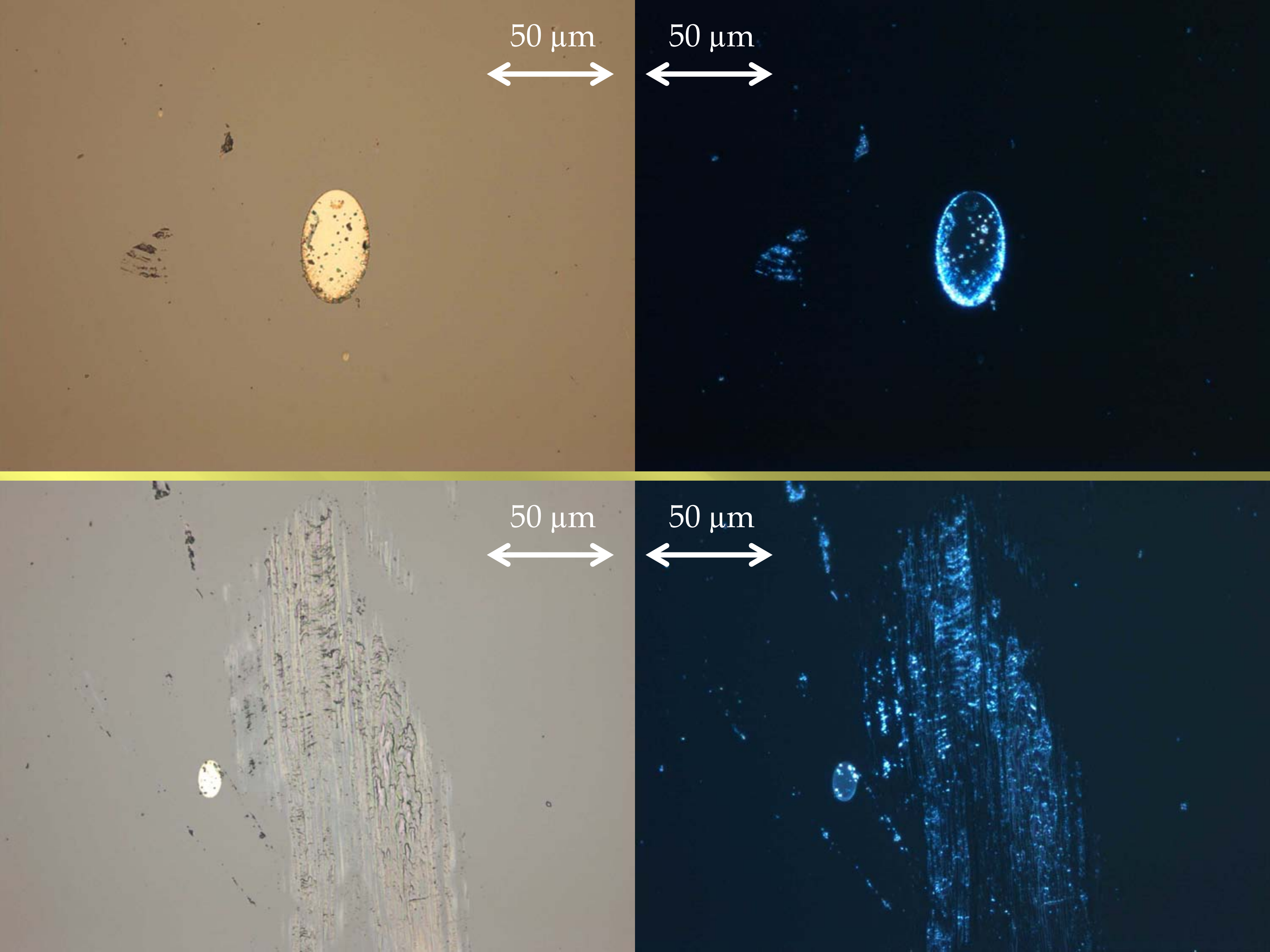

50 µm
2/8/2007, 2:36 PM
60107 SAP B/C Array


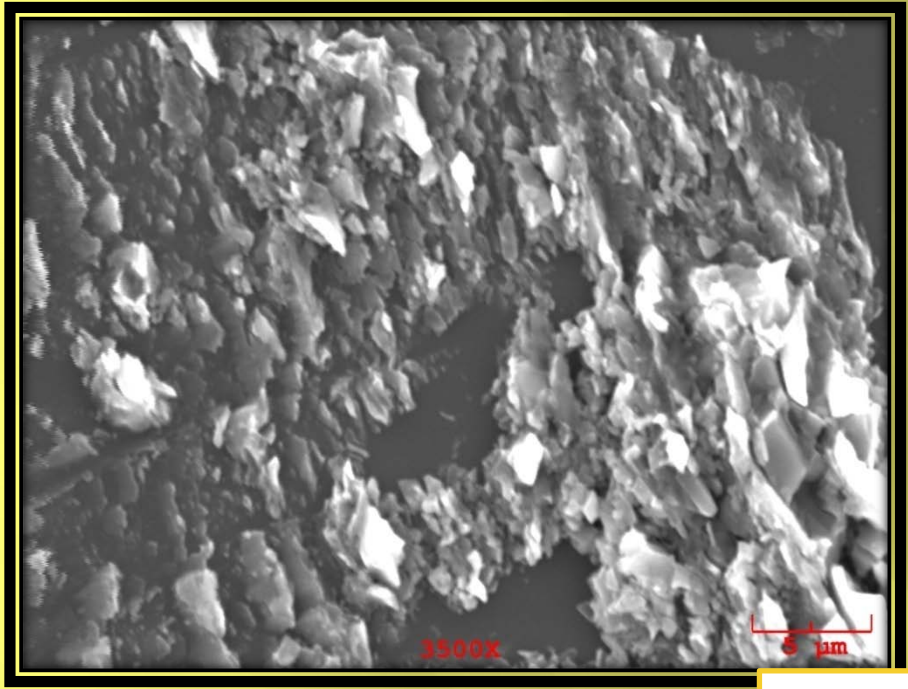

50 µm
2/8/2007, 2:40 PM
60107 SAP B/C array

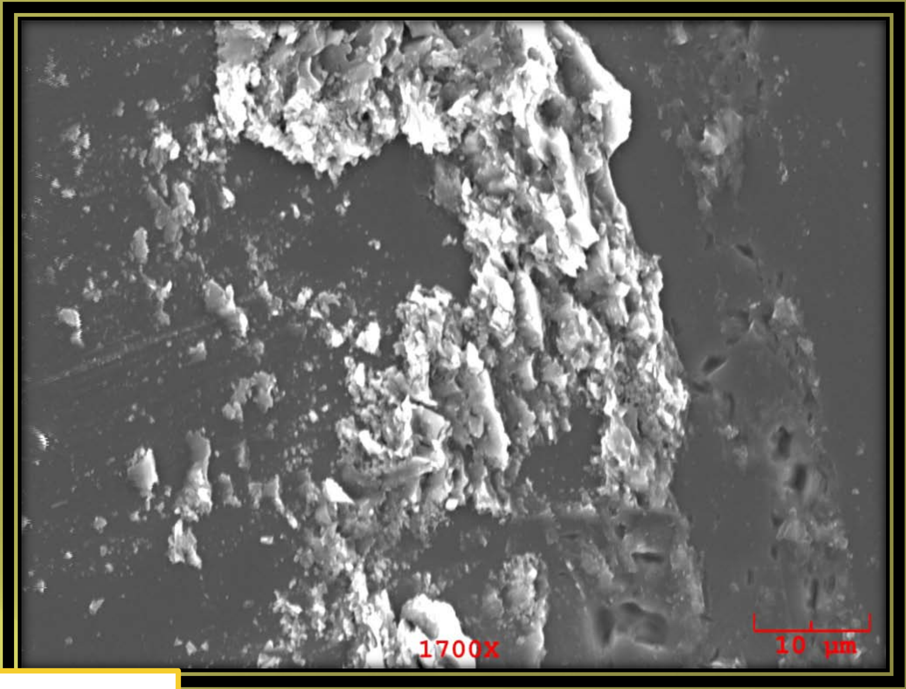

50 µm
2/8/2007, 2:43 PM
60107 SAP B/C array



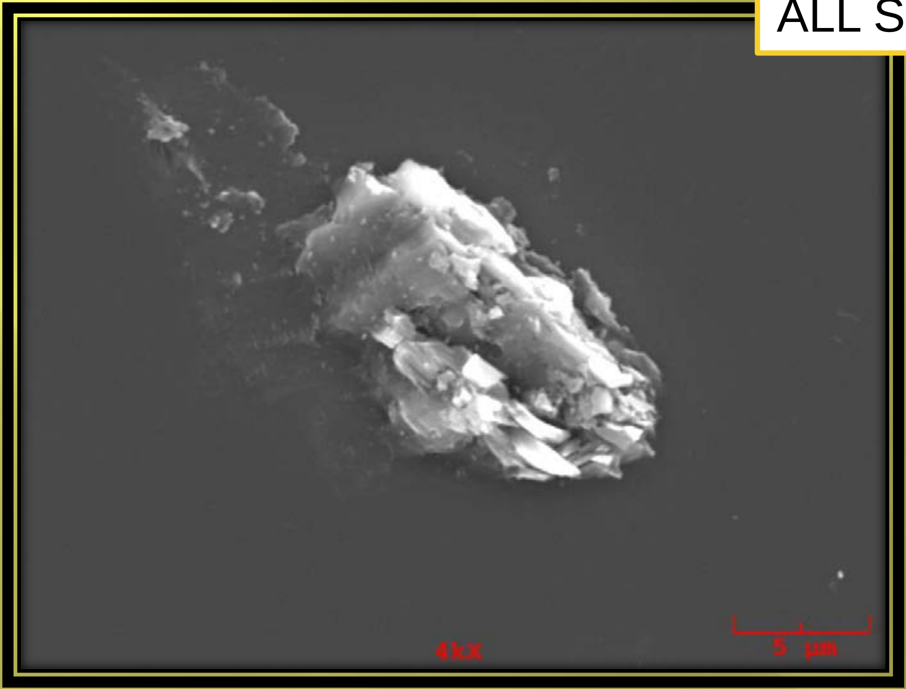


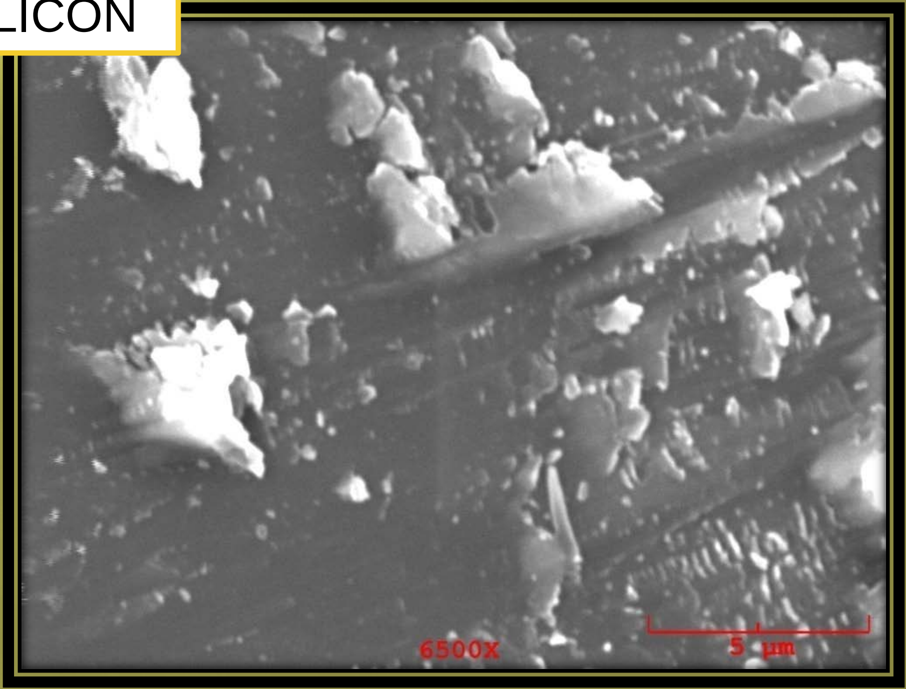

50 μm
50 μm
50 μm
50 μm

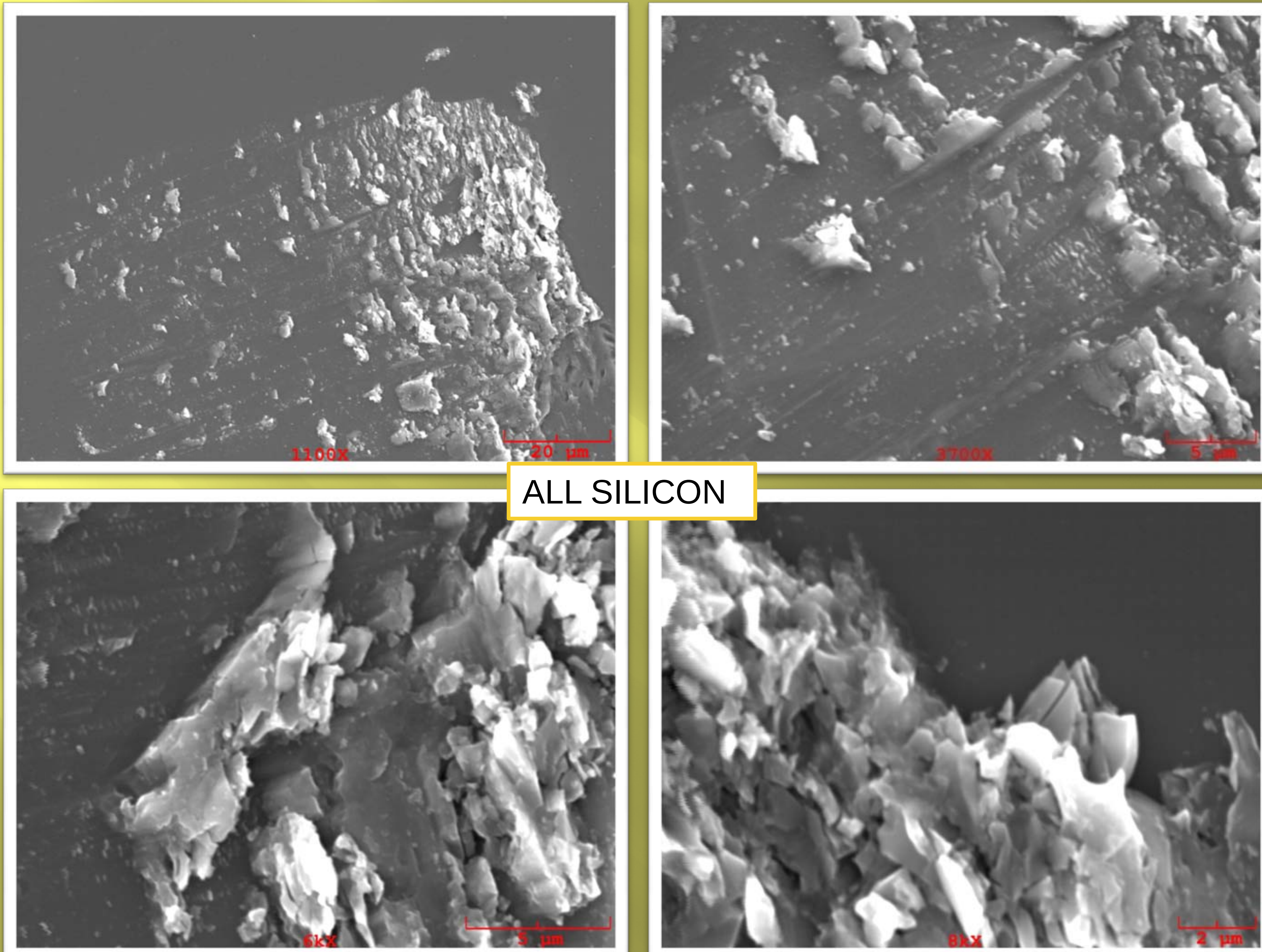




ALL SILICON







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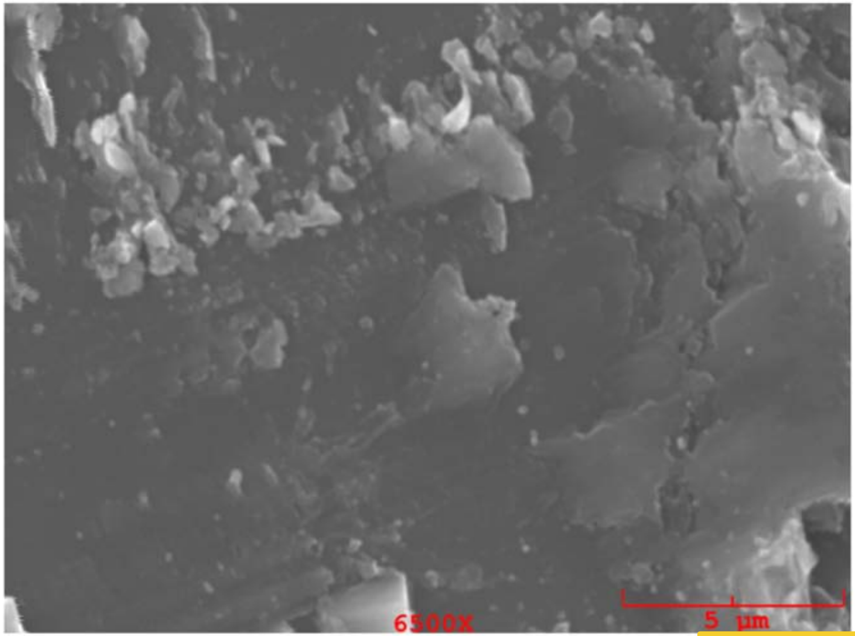

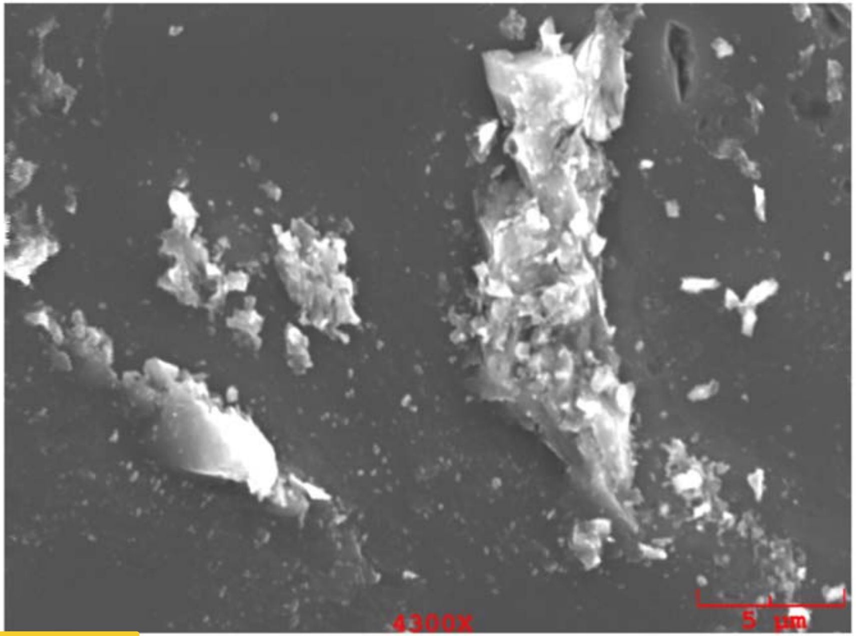

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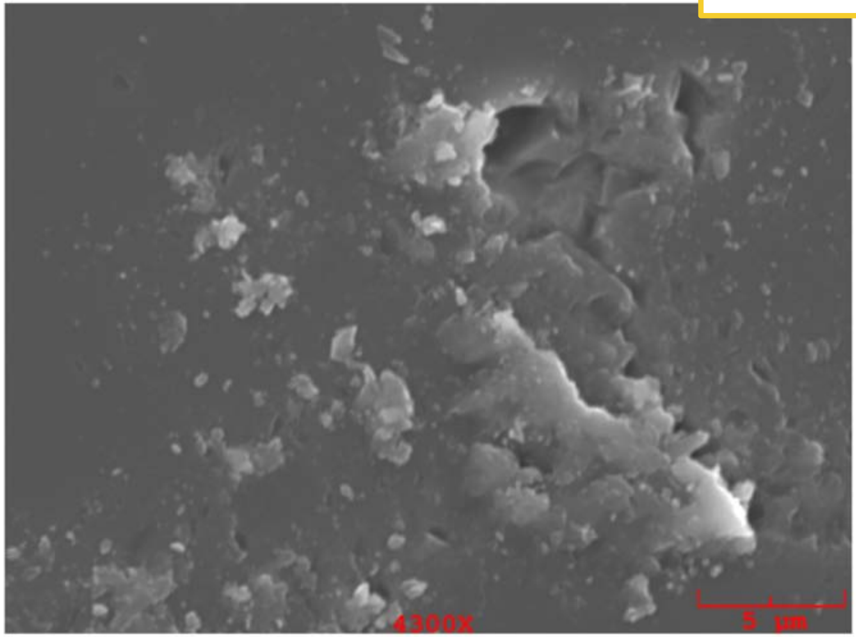
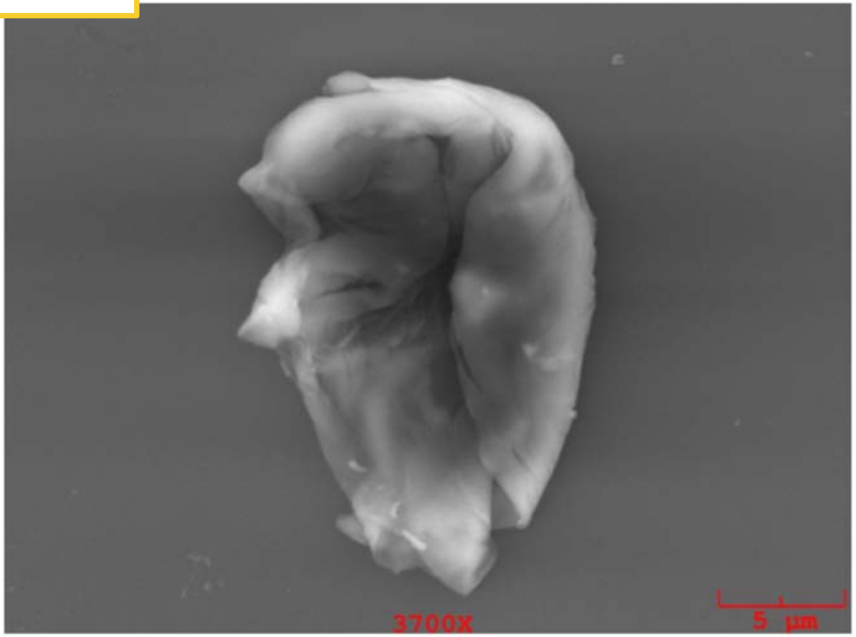
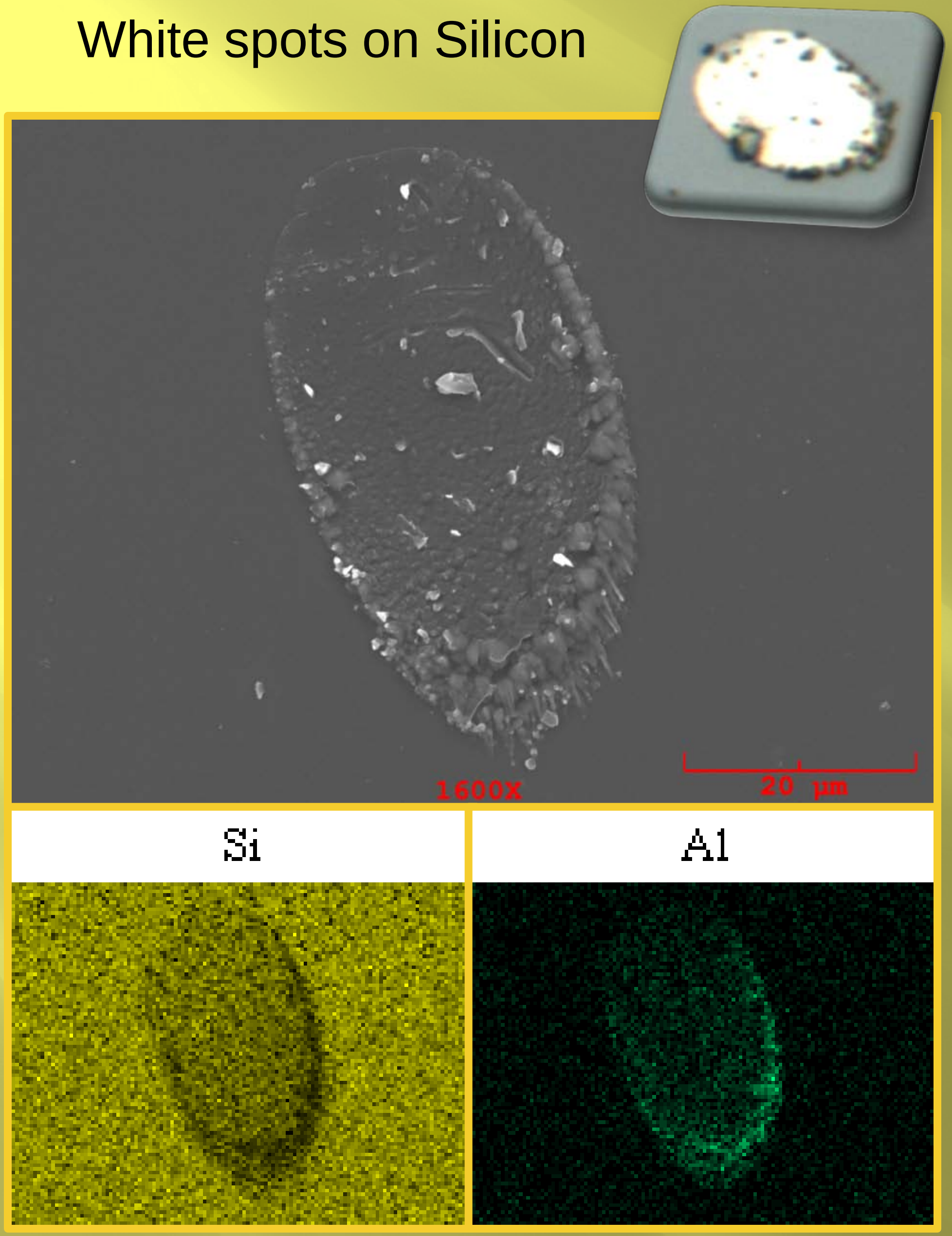




Possible Contamination Sources from the SRC

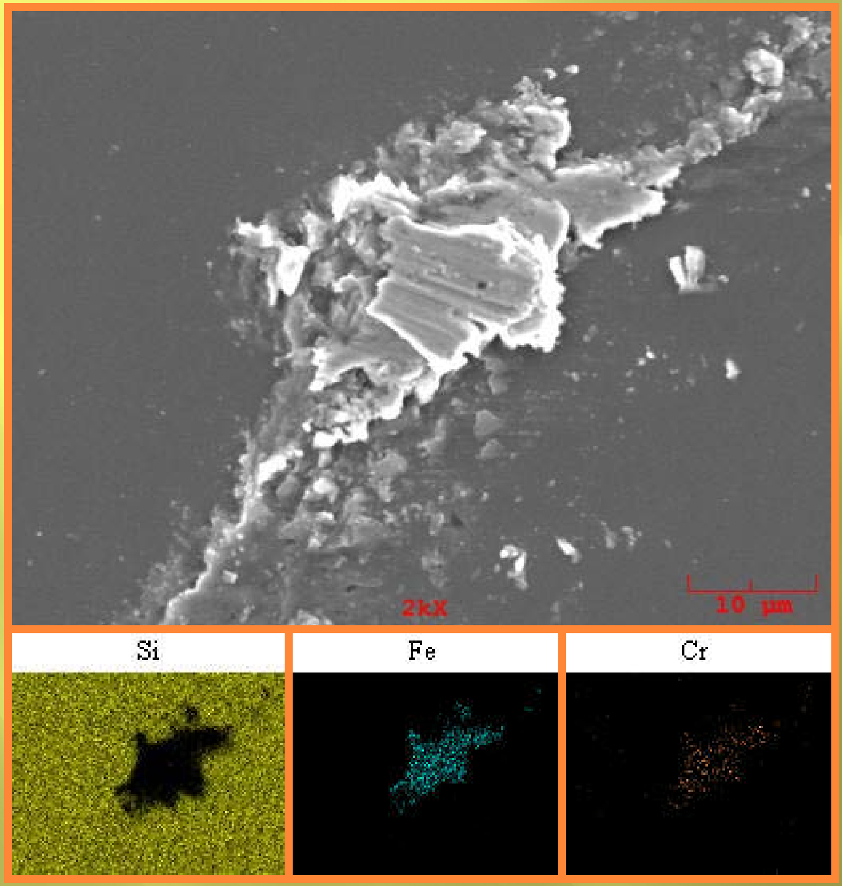
White spots on Silicon

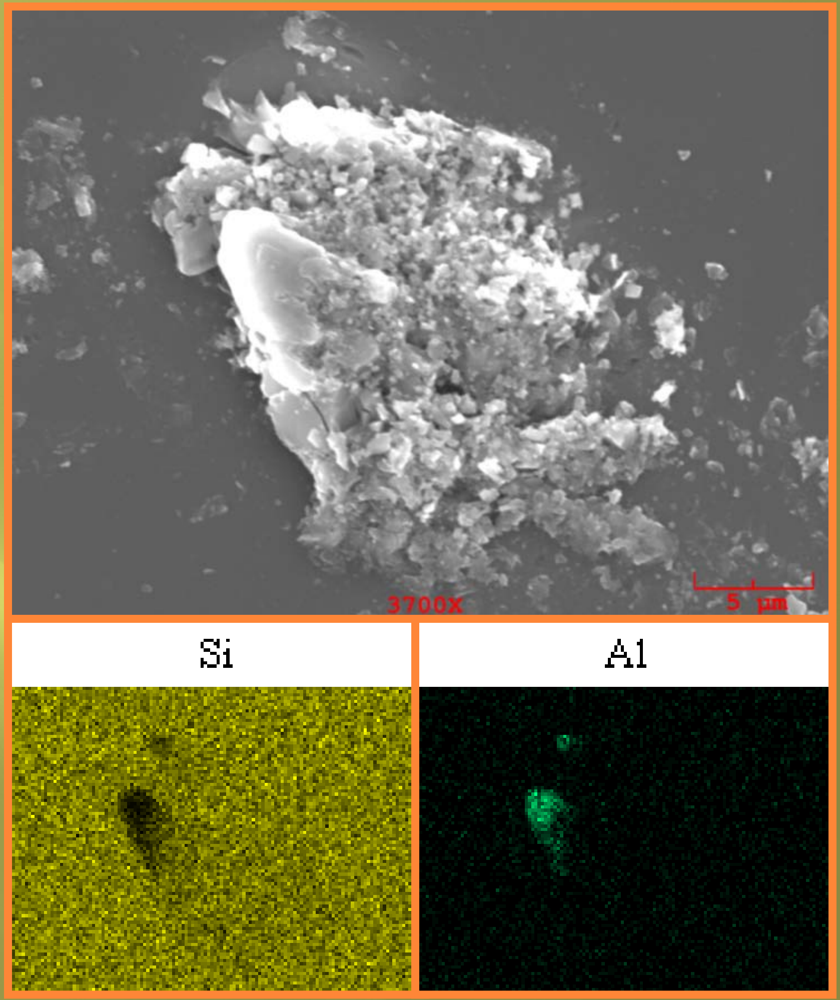


Possible Contamination Source?

Al-arc spray on the C-C ablator

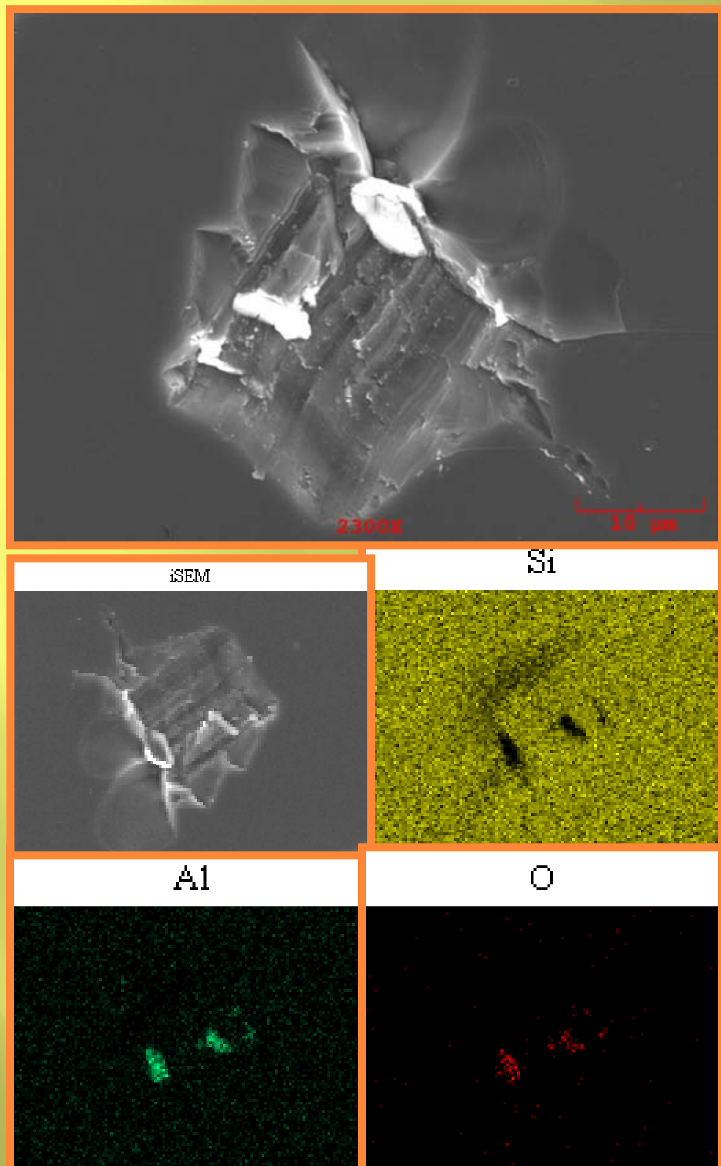
Stainless Steel Contamination

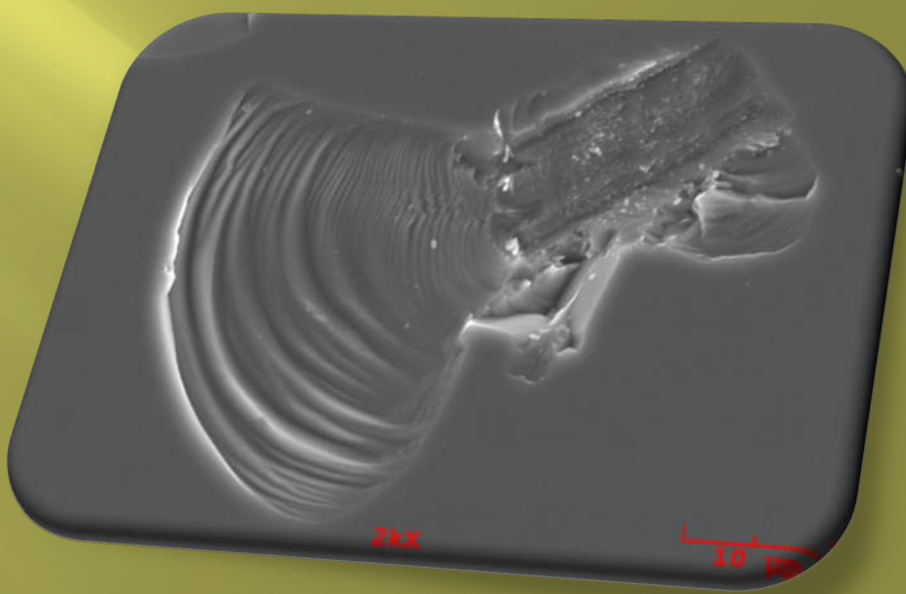




Pure Aluminum Contamination

Scrapes and gouge with fused particles of Al and Al_2O_3 on a Silicon Substrate 60458





Contamination Remaining After UPW/Megasonic Cleaning

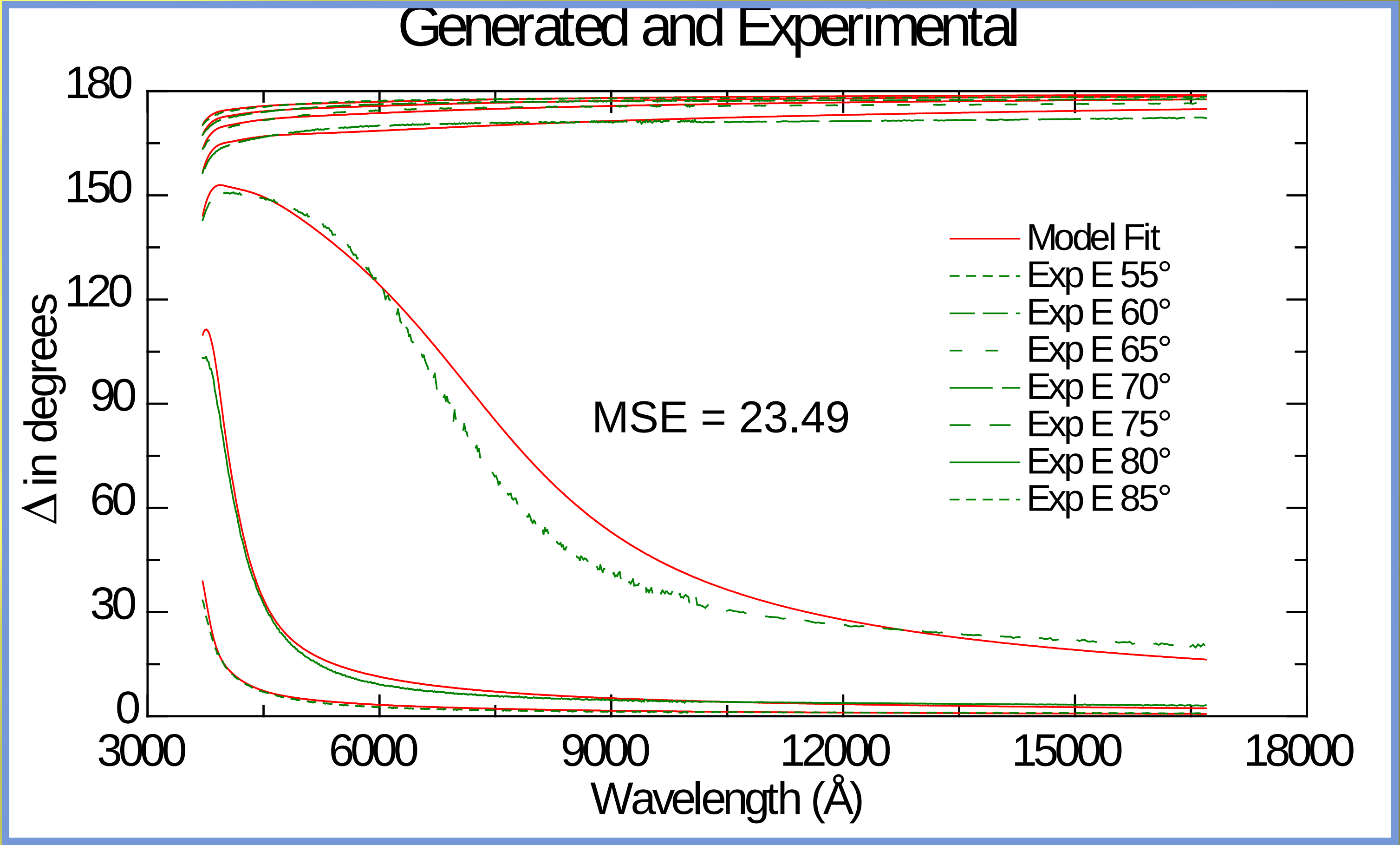
- 92% pure Silicon contamination with no other elements in a randomly selected 0.5 mm^2 area. The vast majority of contamination is Silicon.
- Remaining Si contamination seems to be fused to the Si substrate.
- Sources of Si are possibly from solar wind collectors and SRC super-light ablator (SLA) filled with soda-lime glass.
- Other contamination on 60458 include pure Al, Al_2O_3, and stainless steel.
- Contamination sources for Al and Al_2O_3 are possibly from SLA honeycomb, array frames, and other parts of the SRC.

Spectroscopic Ellipsometer

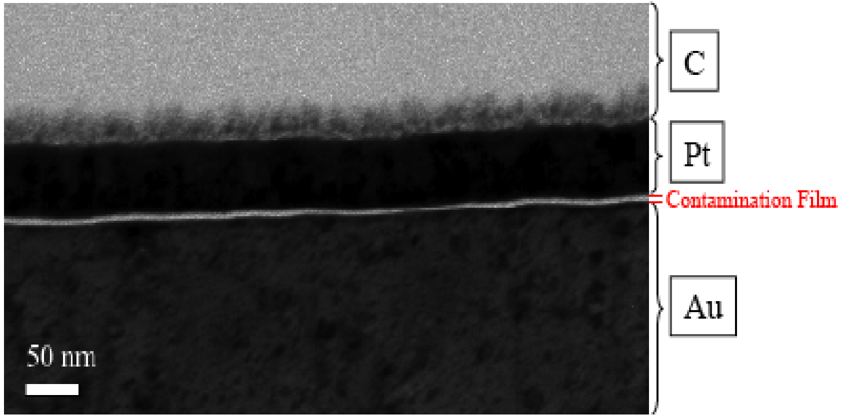
Characterize Molecular Thin -films

Sample 60208 Flown Genesis Si B/C array

1	sio2_jaw	36.13 Å
0	si_jaw	1 mm

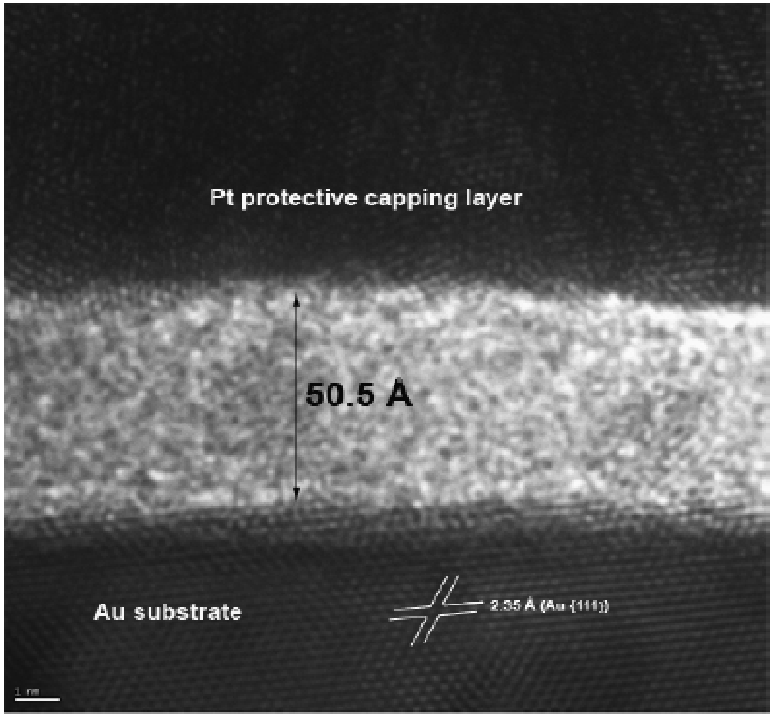


Note that the delta parameter is the difference in the phase of the measured sample between the p- and s-polarized pseudo-Fresnel reflection coefficient at the angle of incidence and a given wavelength



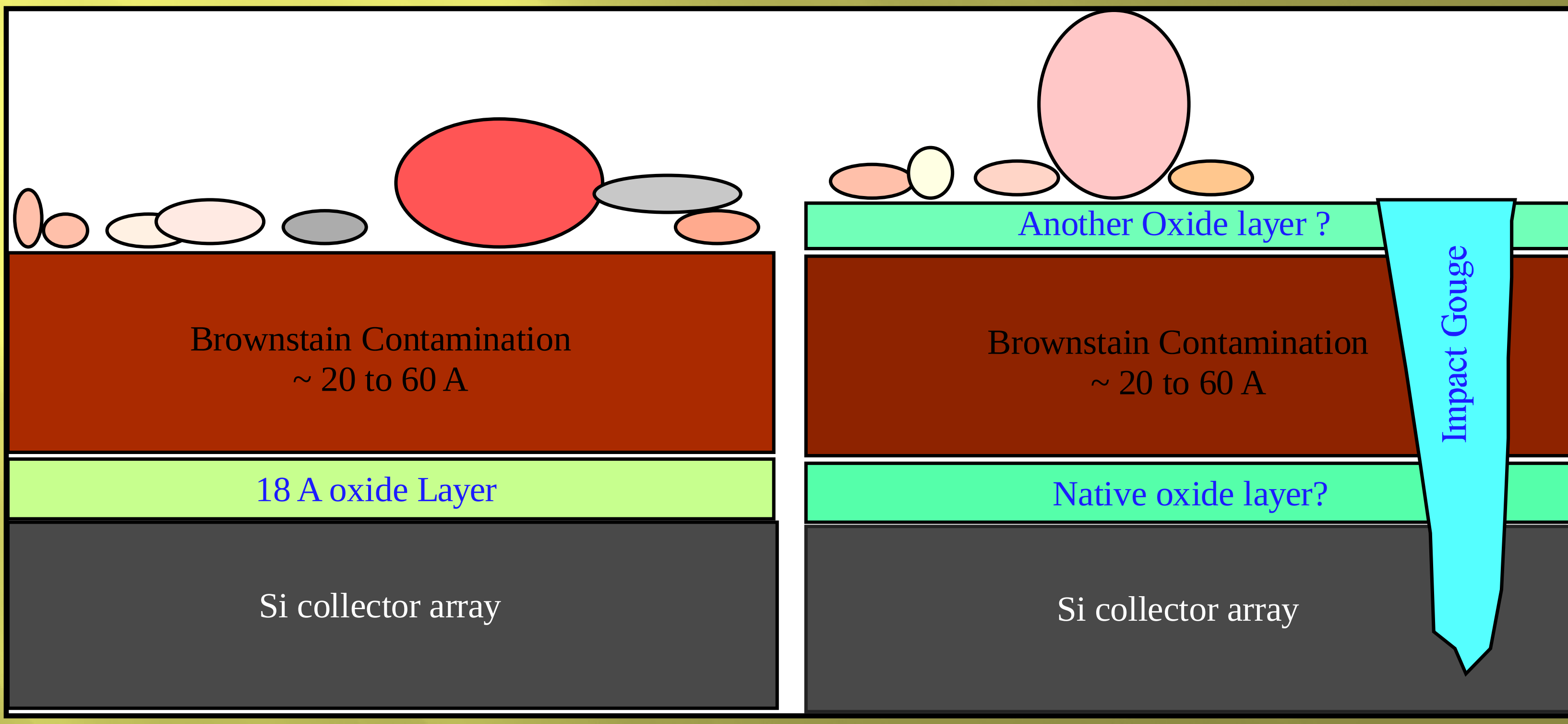
Bright-field micrograph of the FIB lift-out membrane

The Famous Brown Stain Visible on the Gold Foil



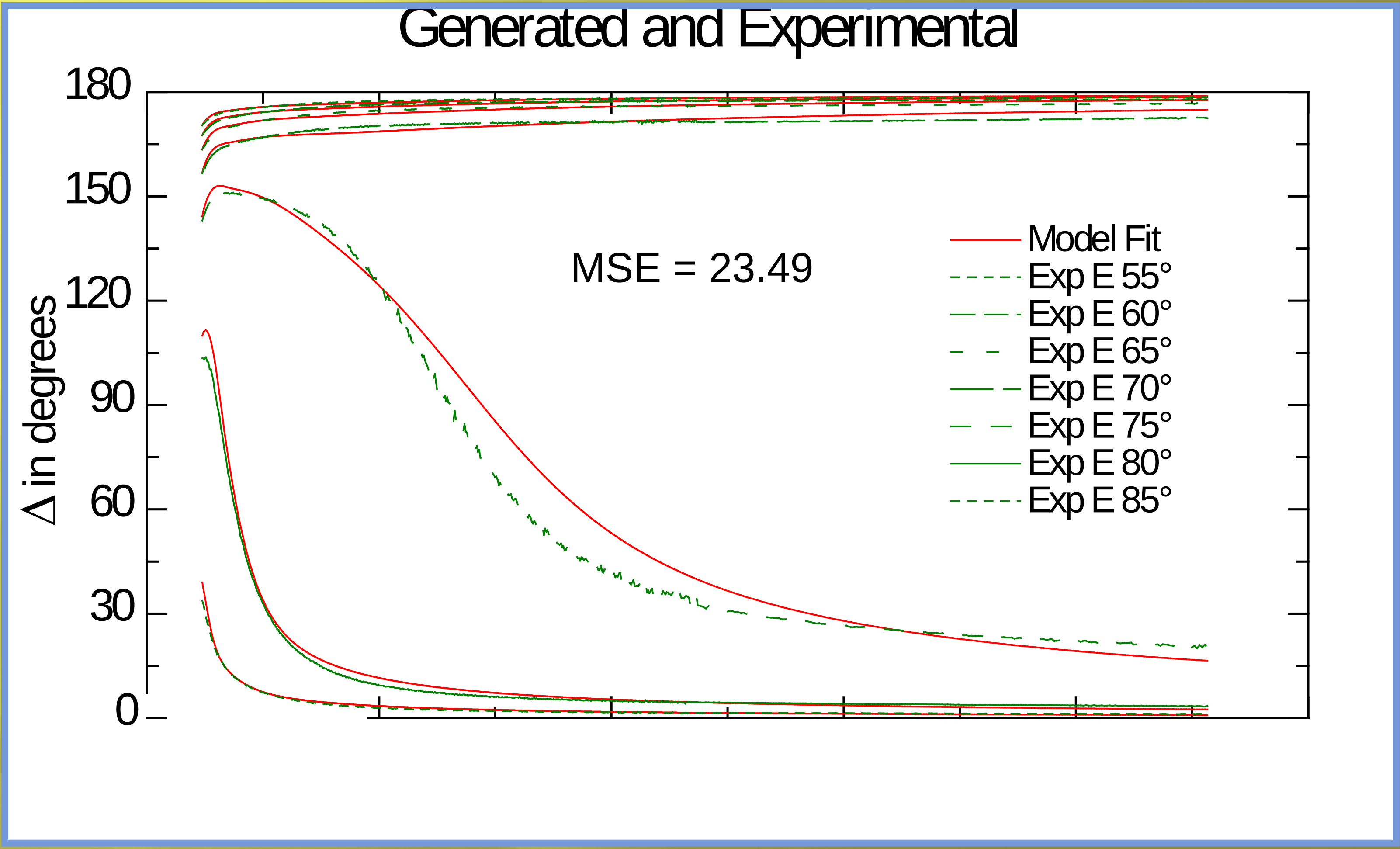
HRTEM micrograph showing the contamination film between the Pt protective capped layer and the Genesis Au-foil. Thickness of the contamination film is 50.5 Å.

If Brown Stain Existed: Two possible Ellipsometry Models (2005-2006)

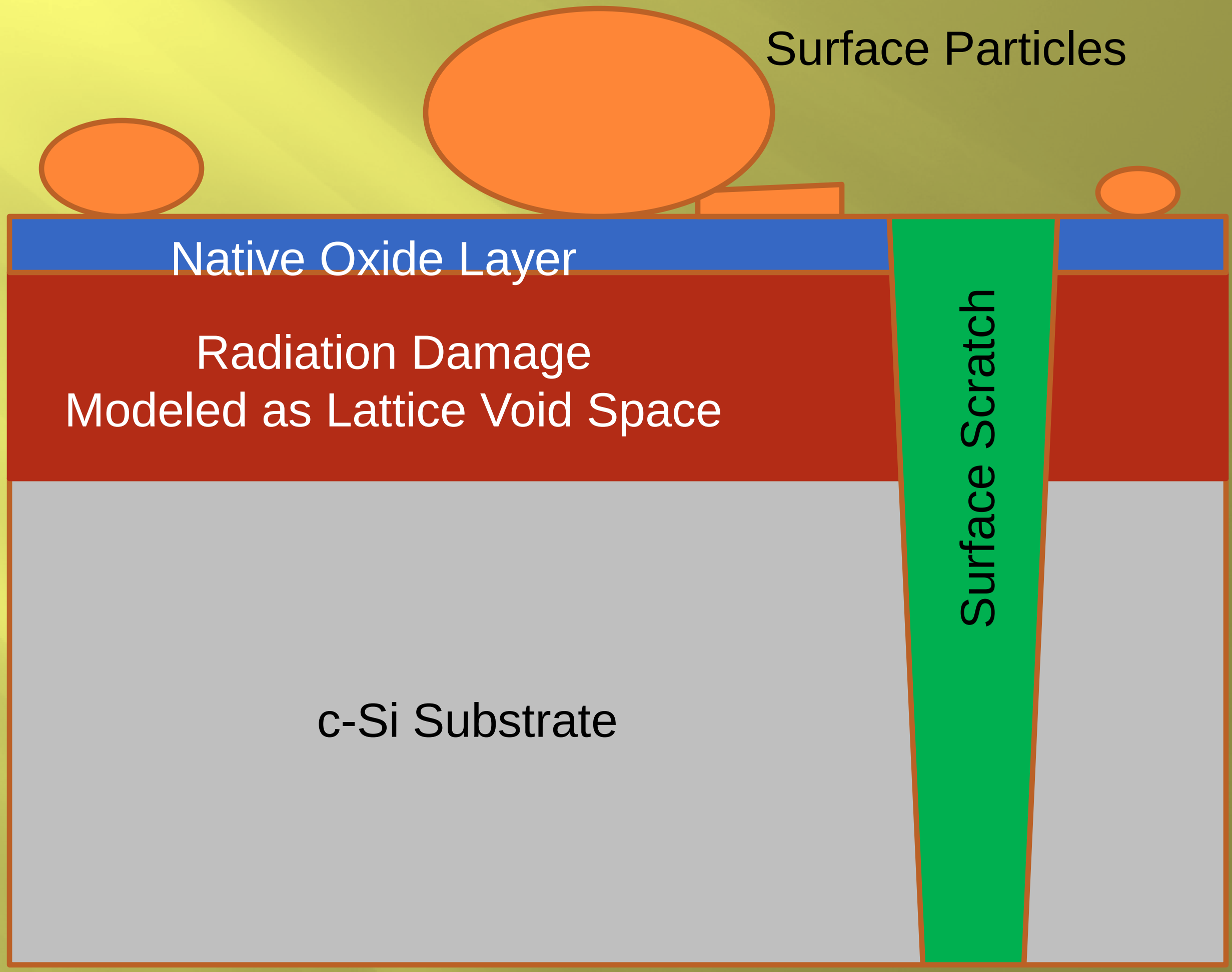


Sample 60208 Flown Genesis Si B/C array

2	cauchy	0.00 Å
1	sio2_jaw	36.14 Å
0	si_jaw	1 mm

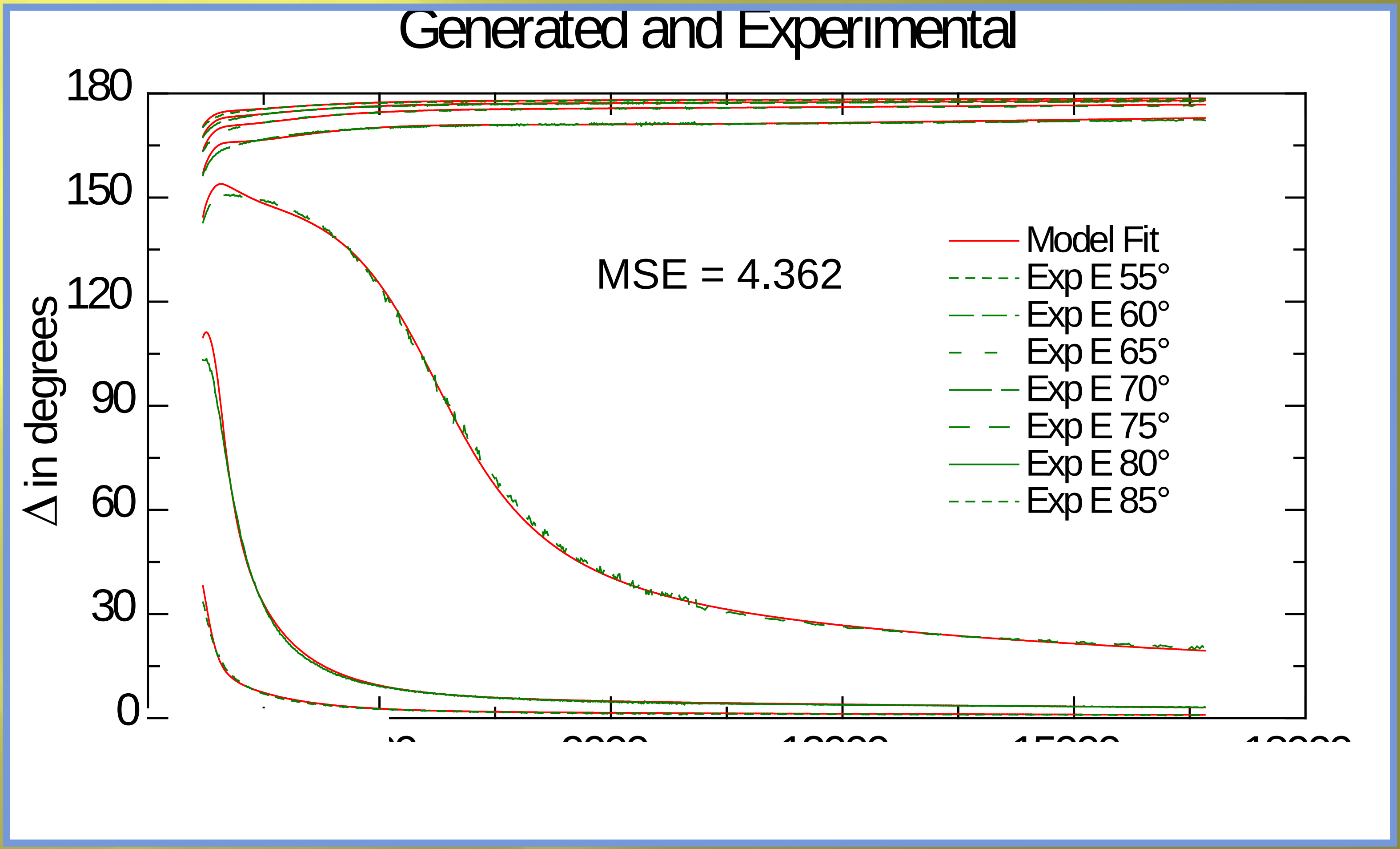


If Brown Stain was not present:
Ellipsometry Model for Radiation Damage



Sample 60208 Flown Genesis Si B/C array

2	sio2_jaw	34.55 Å
1	EMA (si_jaw)/1.24% void	609.85 Å
0	si_jaw	1 mm



Sample 60208 Flown Genesis Si B/C array

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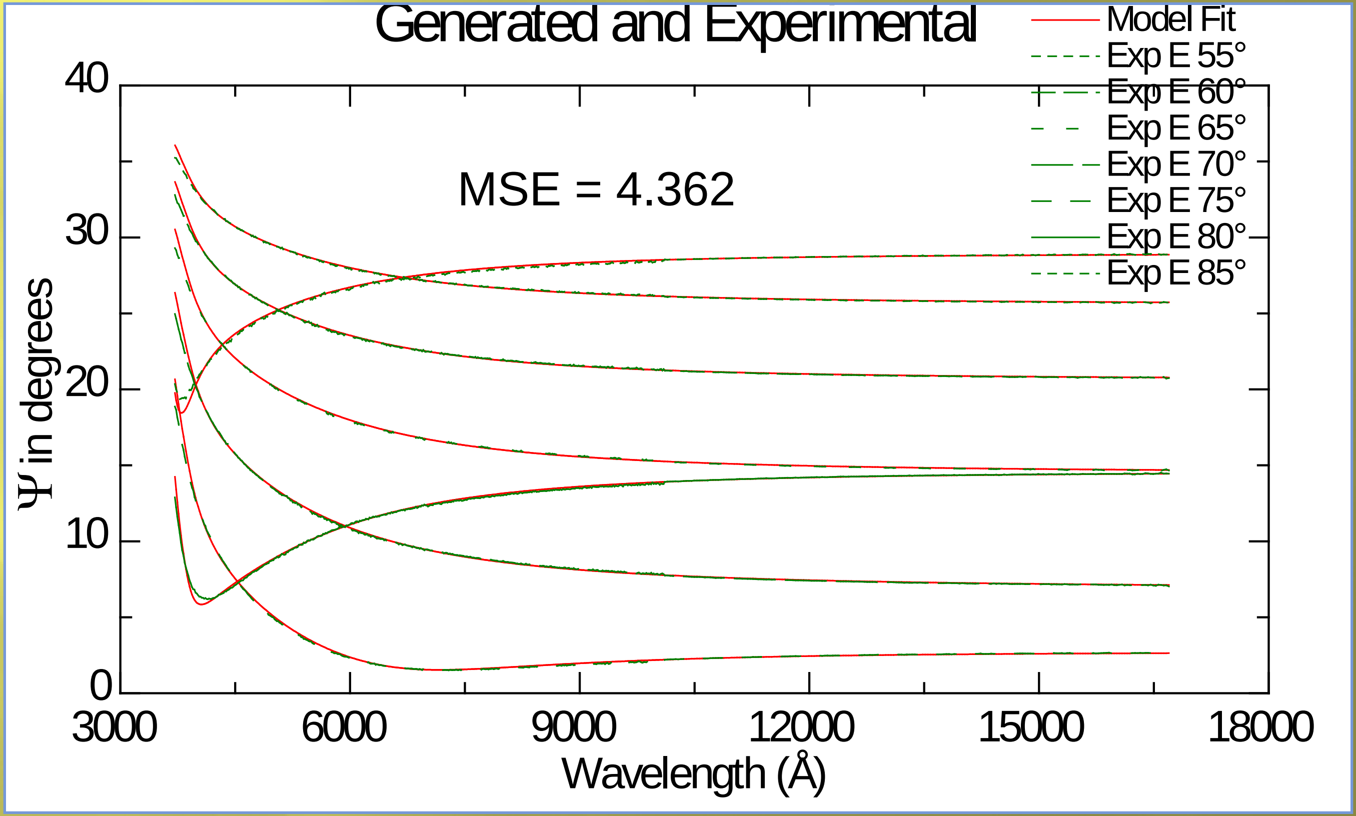
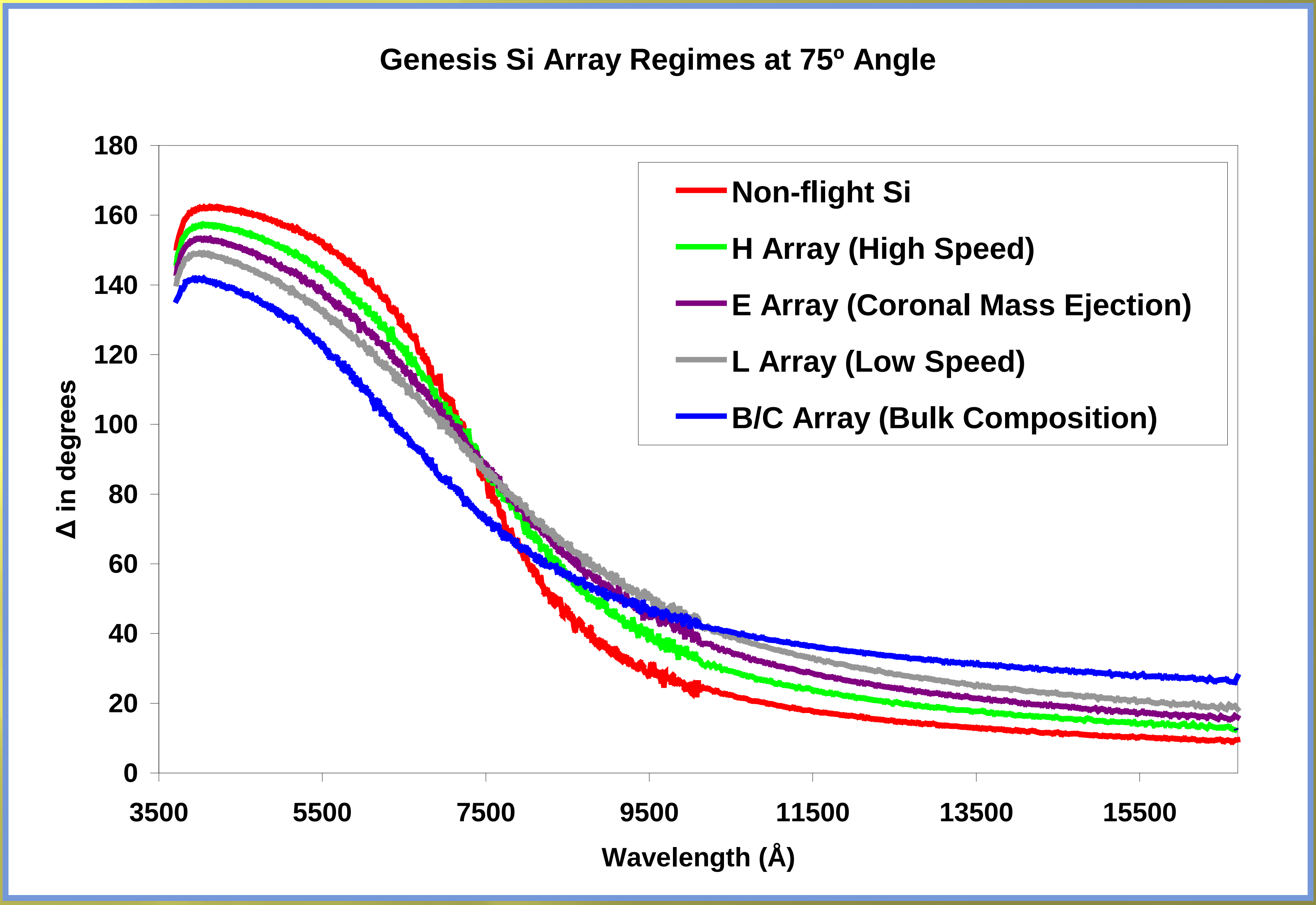


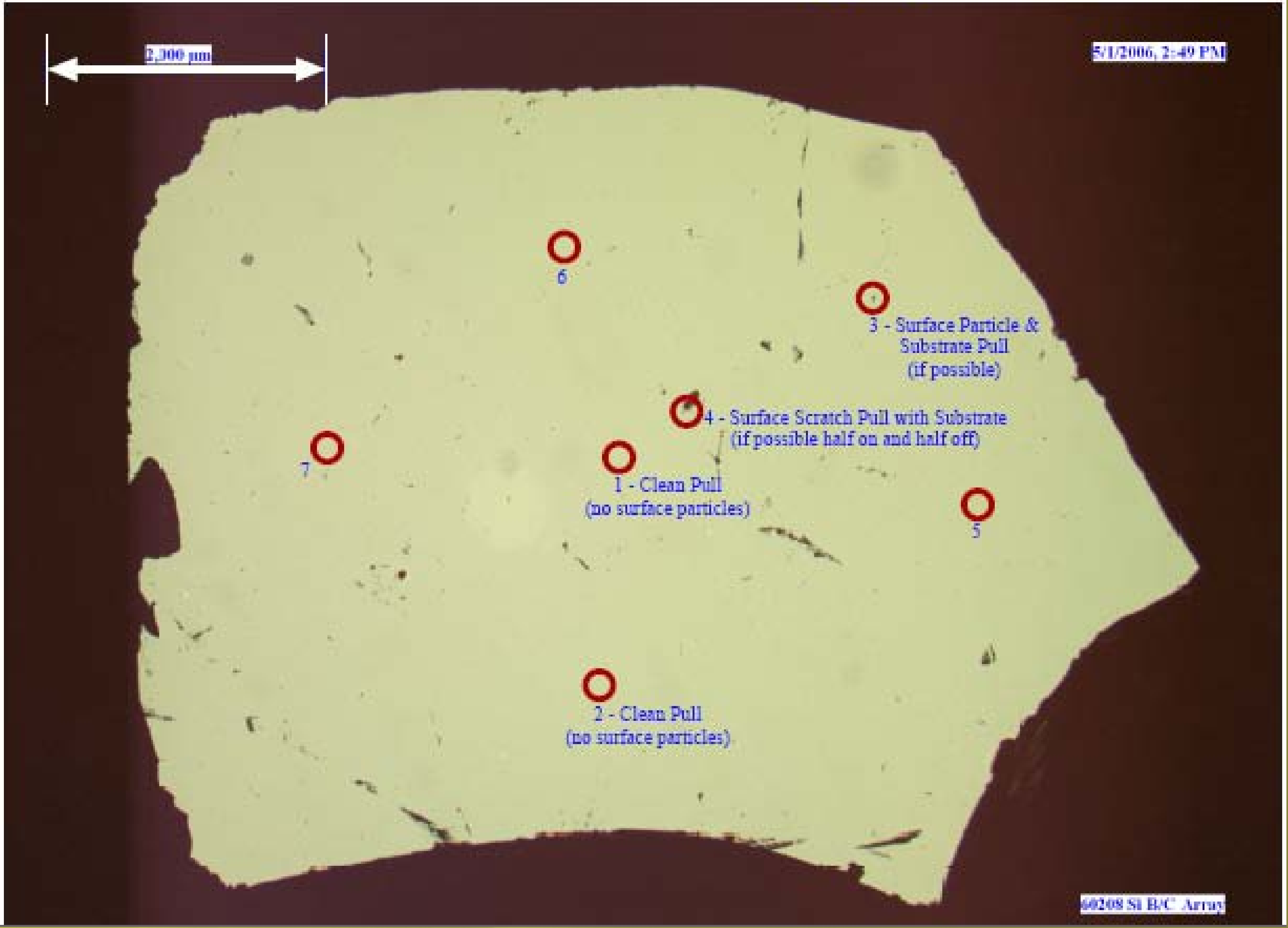
PSI is the magnitude of the ratio of the p- to s- direction pseudo-Fresnel reflection coefficient of the sample at the given wavelength and angle of incidence.


Genesis Si Array Regimes at 75º Angle
Non-flight Si
H Array (High Speed)
E Array (Coronal Mass Ejection)
L Array (Low Speed)
B/C Array (Bulk Composition)
Δ in degrees
0
20
40
60
80
100
120
140
160
180
3500
5500
7500
9500
11500
13500
15500
Wavelength (Å)

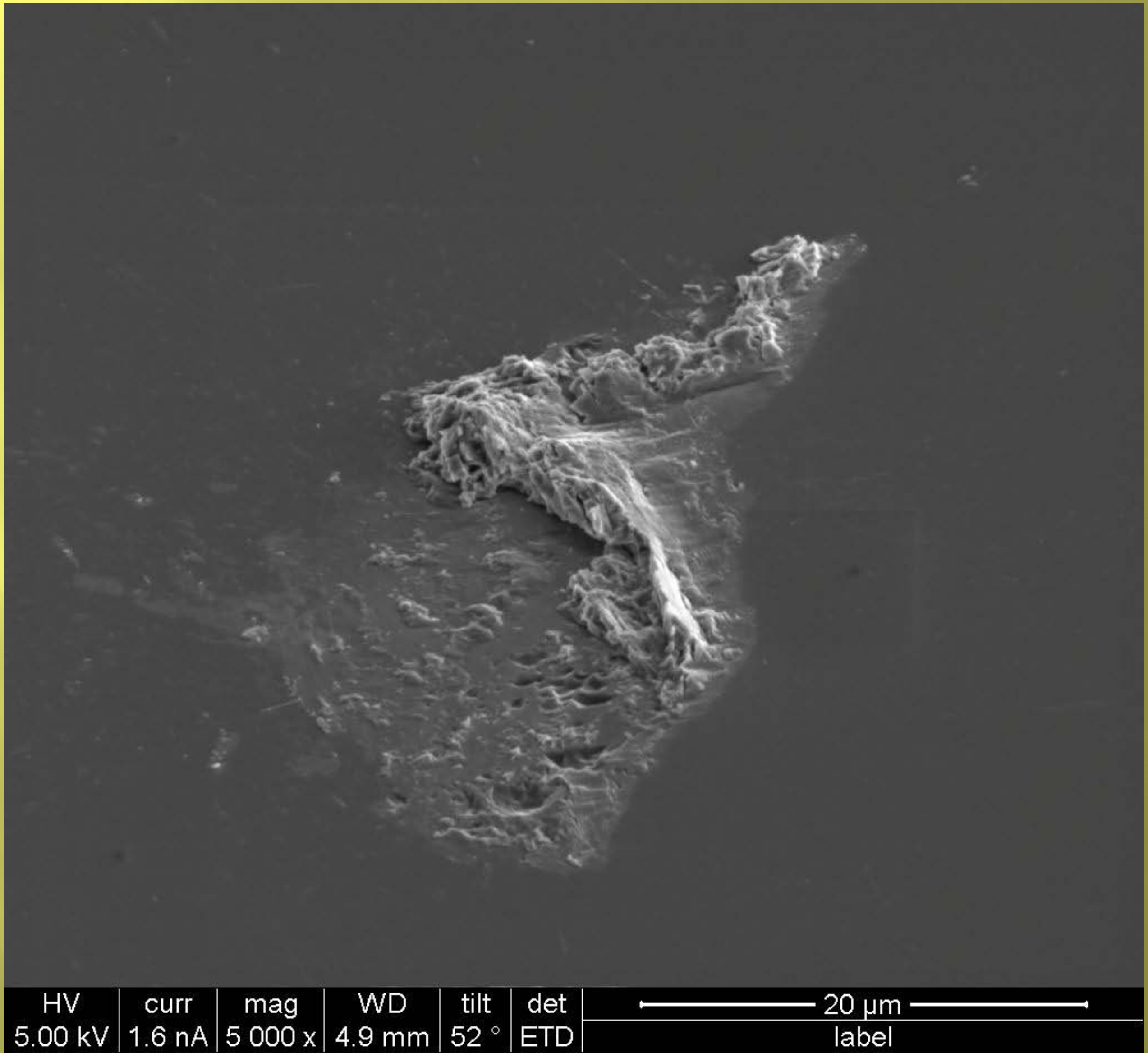
STEM Study Objectives

- Characterize the Brown Stain and any other thin film contamination.
- Characterize (if possible) surface particle contamination and the interaction with the wafer surface.
- Characterize the native oxide layer and verify ellipsometry thickness results.
- Verify ellipsometry EMA layer model for Silicon and substrate alteration thickness.
- Did the Silicon substrate experience lattice alteration during flight?

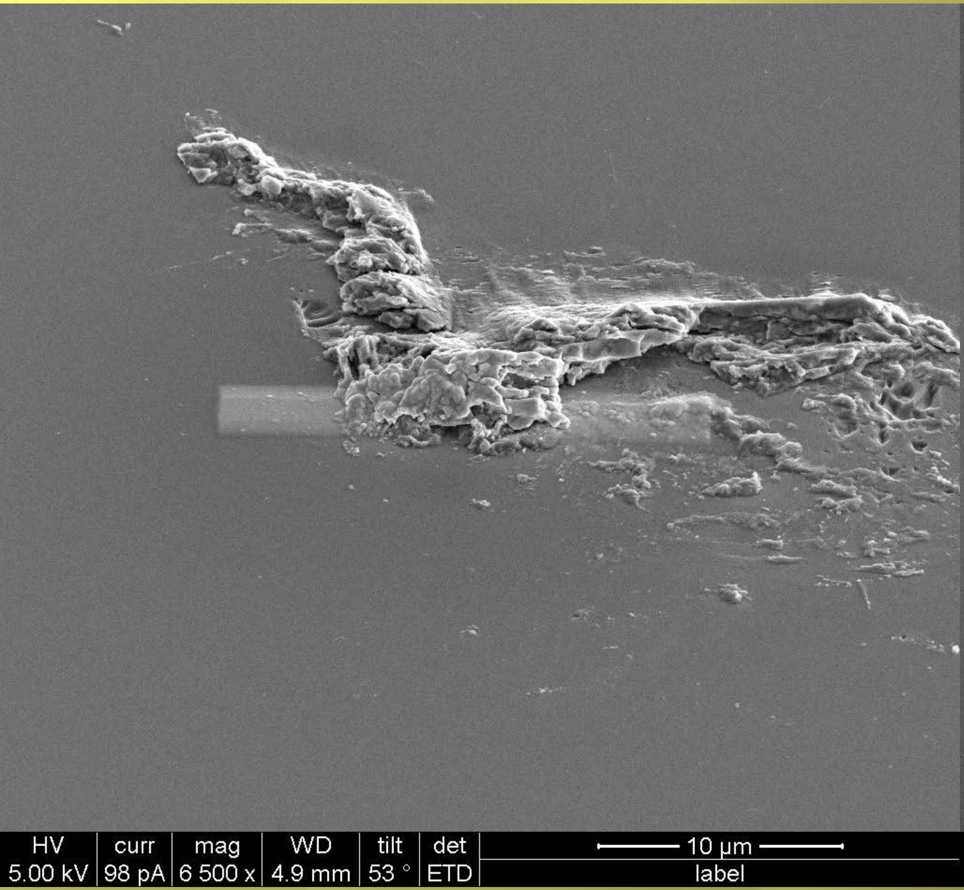
FIB Pull Locations for Stratigraphic Profiles

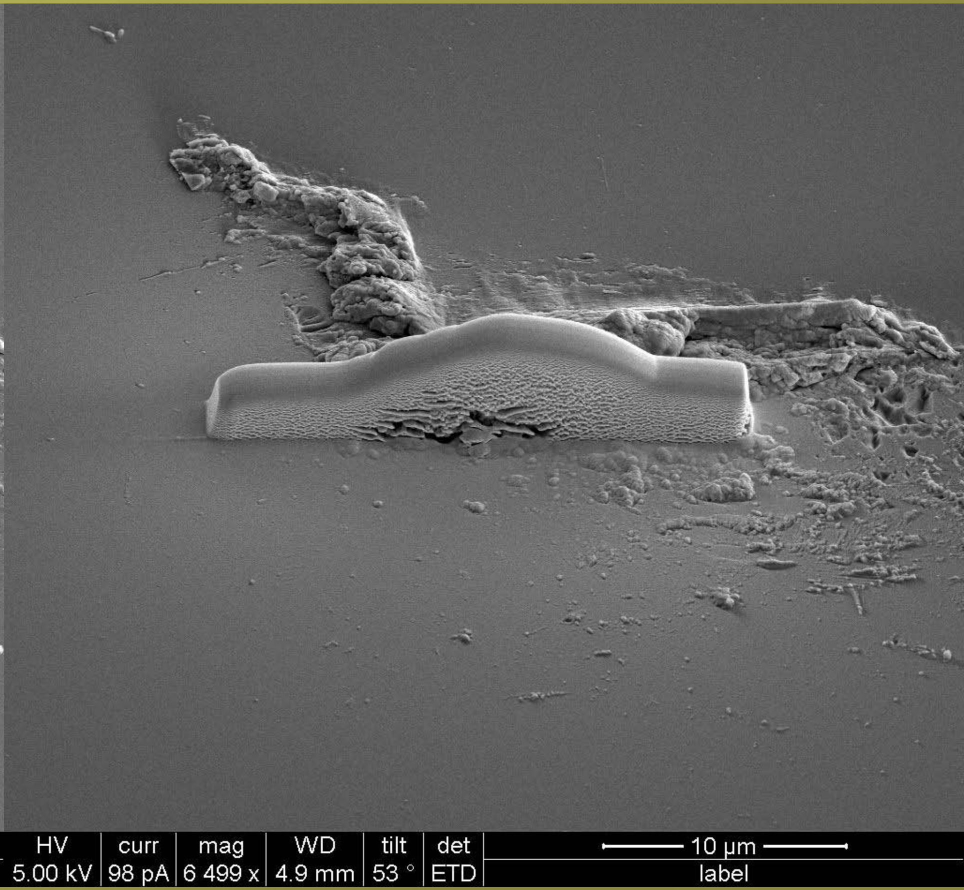


FIB Stratigraphic Cross-section Pull
Sample 60208.4 (Surface particle Pull)

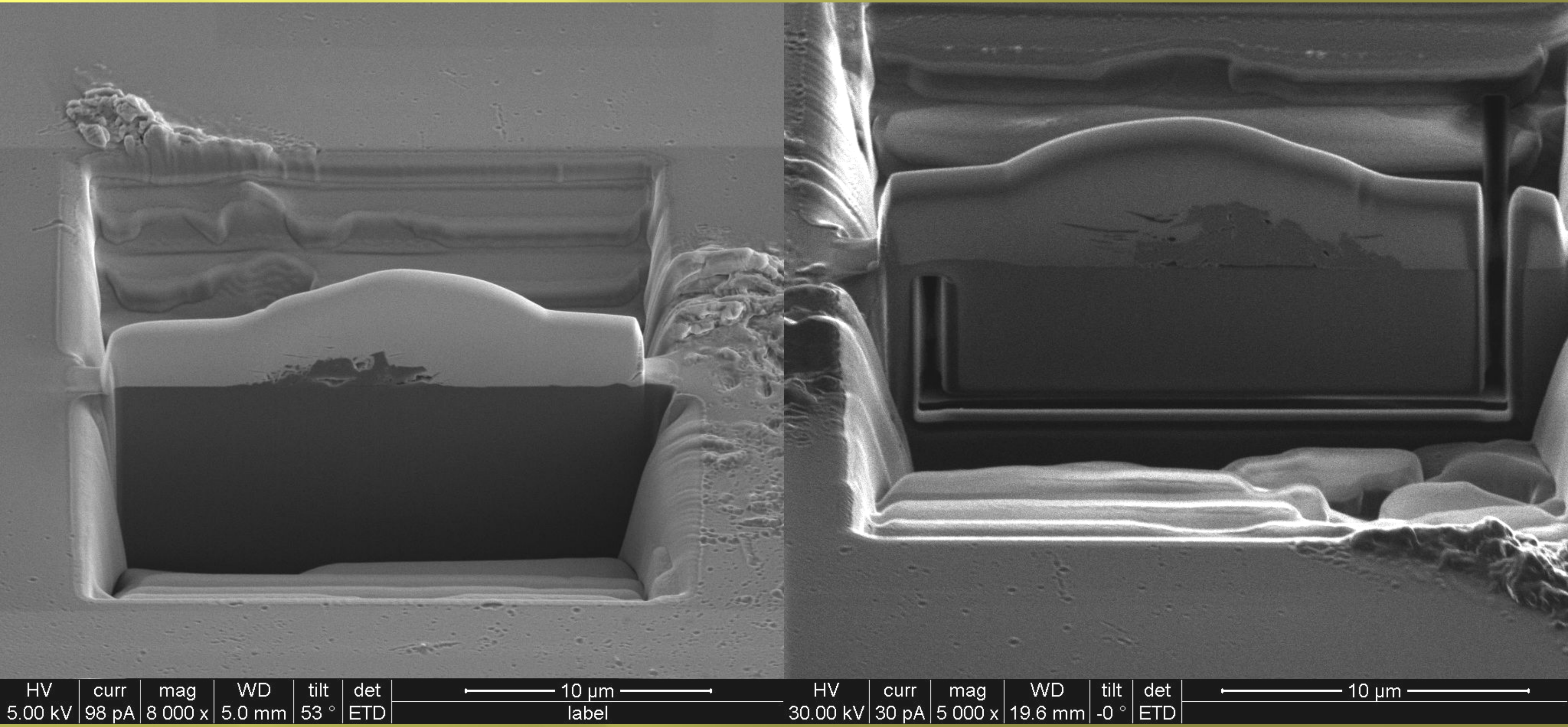


Application of a Platinum Coating for FIB Pull Section

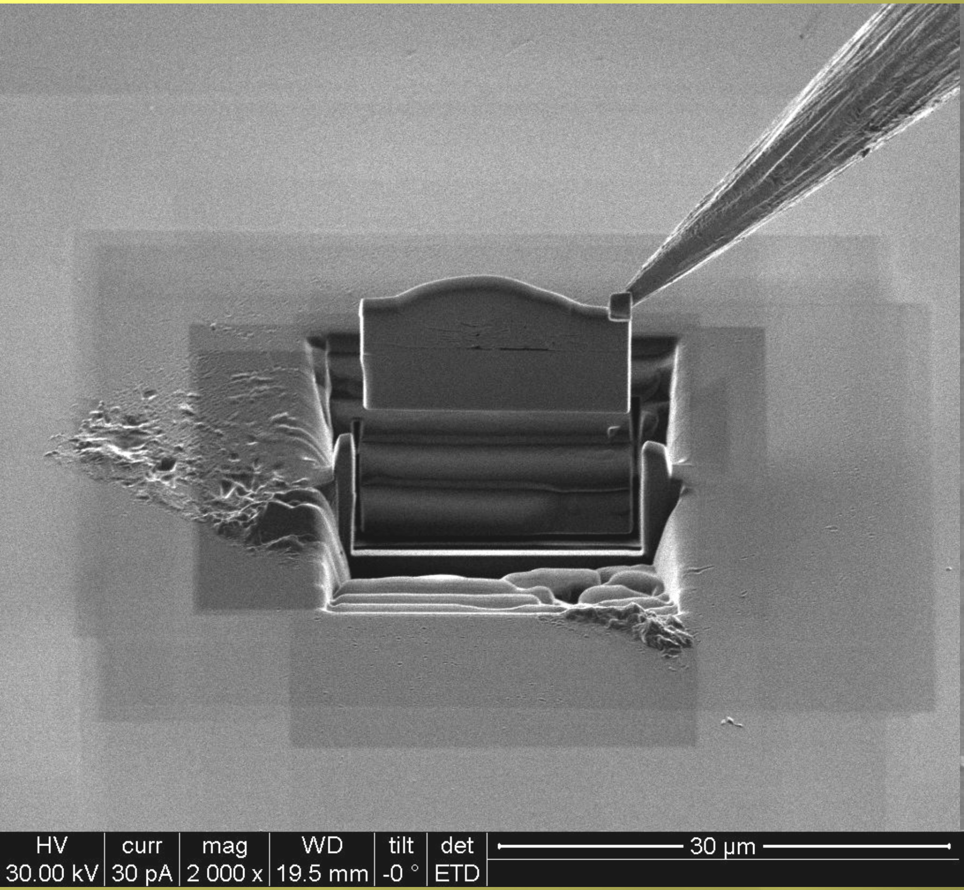


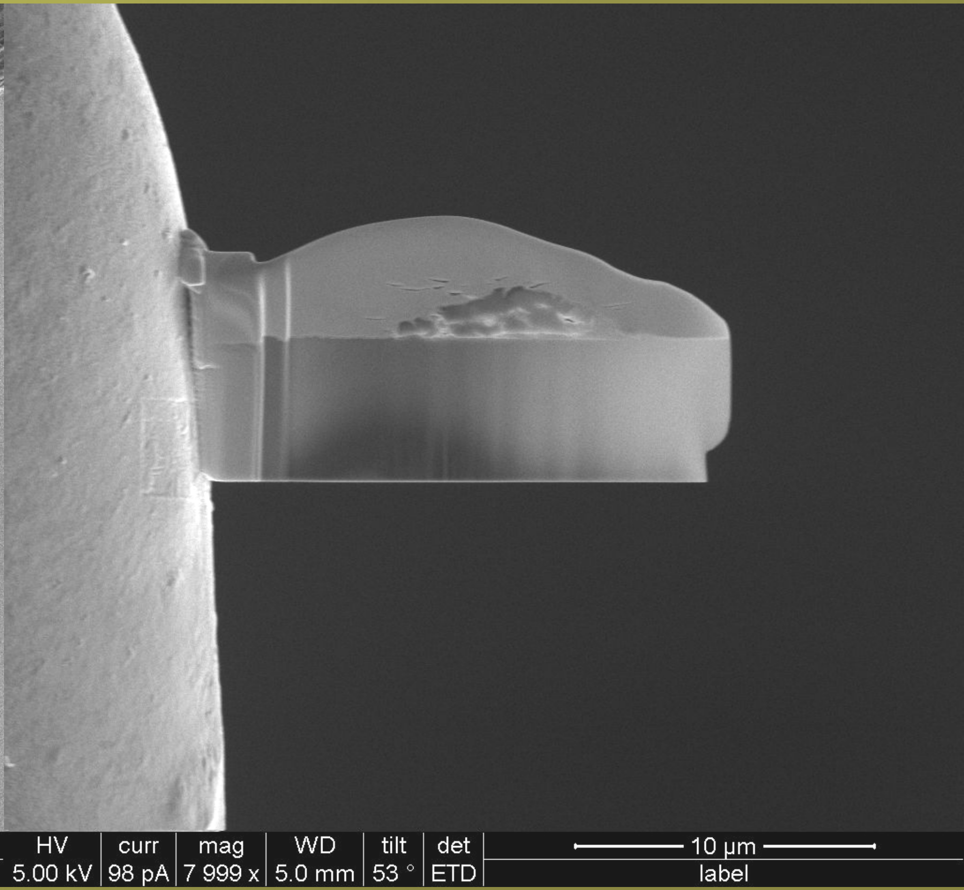


Ion Milling Stratigraphic Cross-section

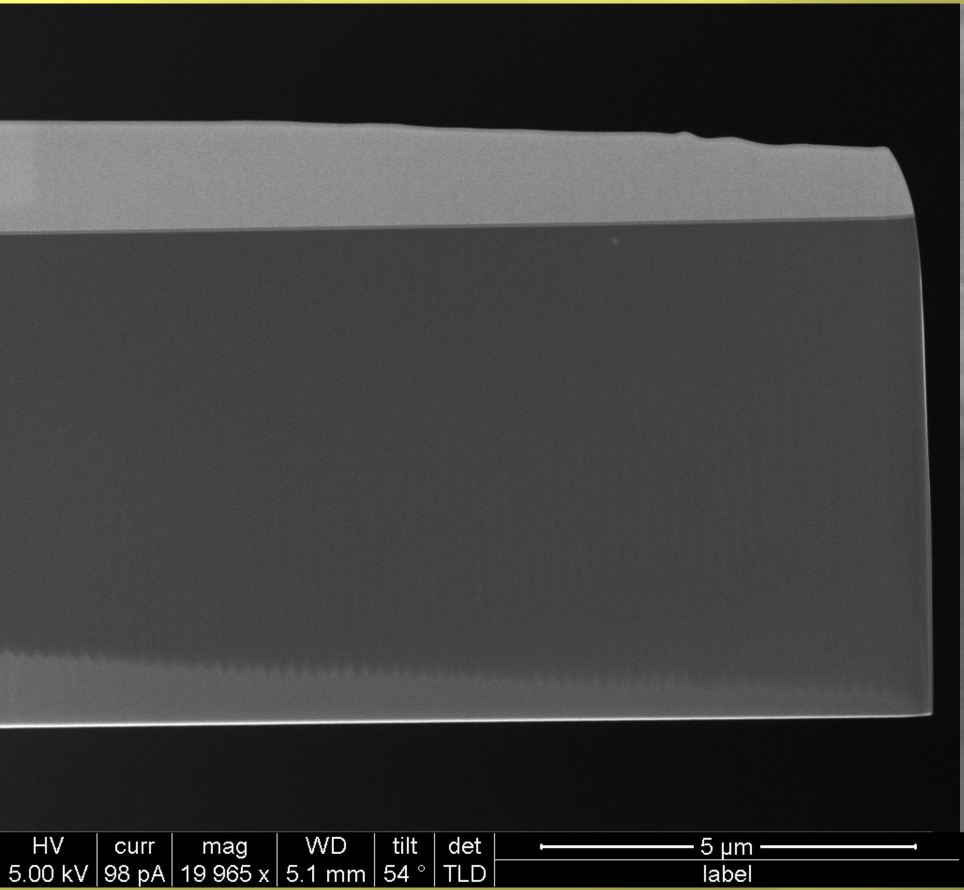


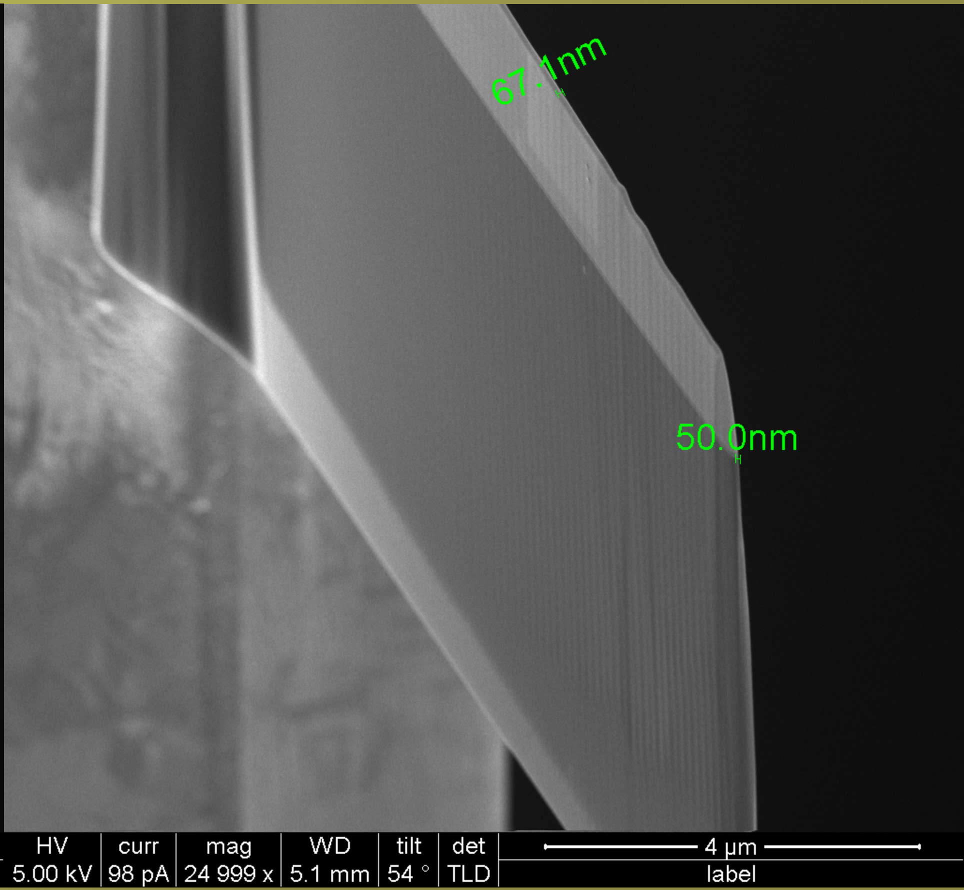
FIB Pull and Mounting Section on TEM Grid





Final Configuration After Thinning
Sample 60208.1
(~ 10 x 6 μm and ~ 60 nm thick)



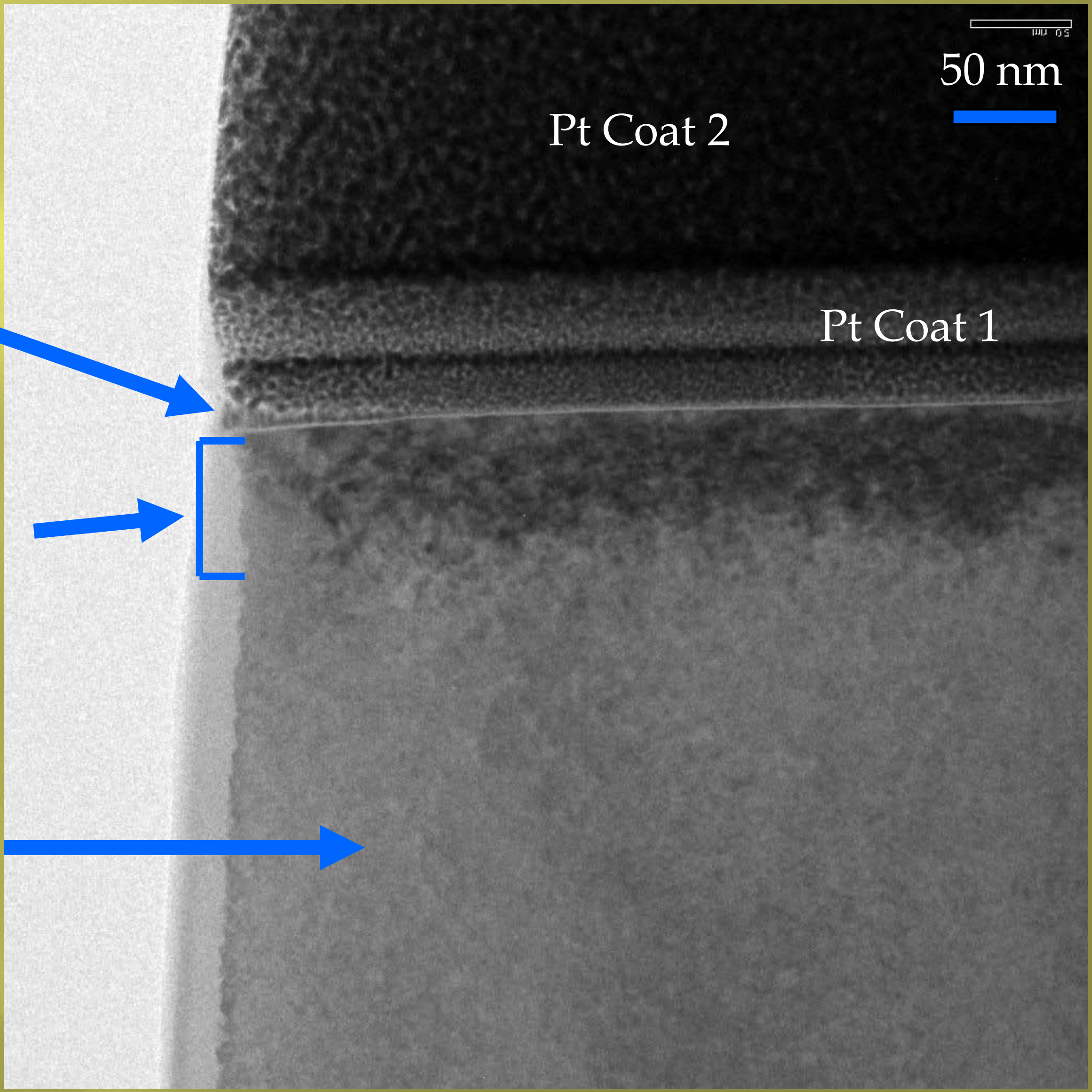


JEOL JEM 2500SE STEM Image of 60208.1 Si B/C Array

Wafer Surface
Native Oxide Layer
SiO_2 = ~ 38 Å

Complex Diffraction Contrast
Radiation Damage Zone
Depth = ~ 610 Å
Range = 60 - 75 nm

Crystalline Si Substrate



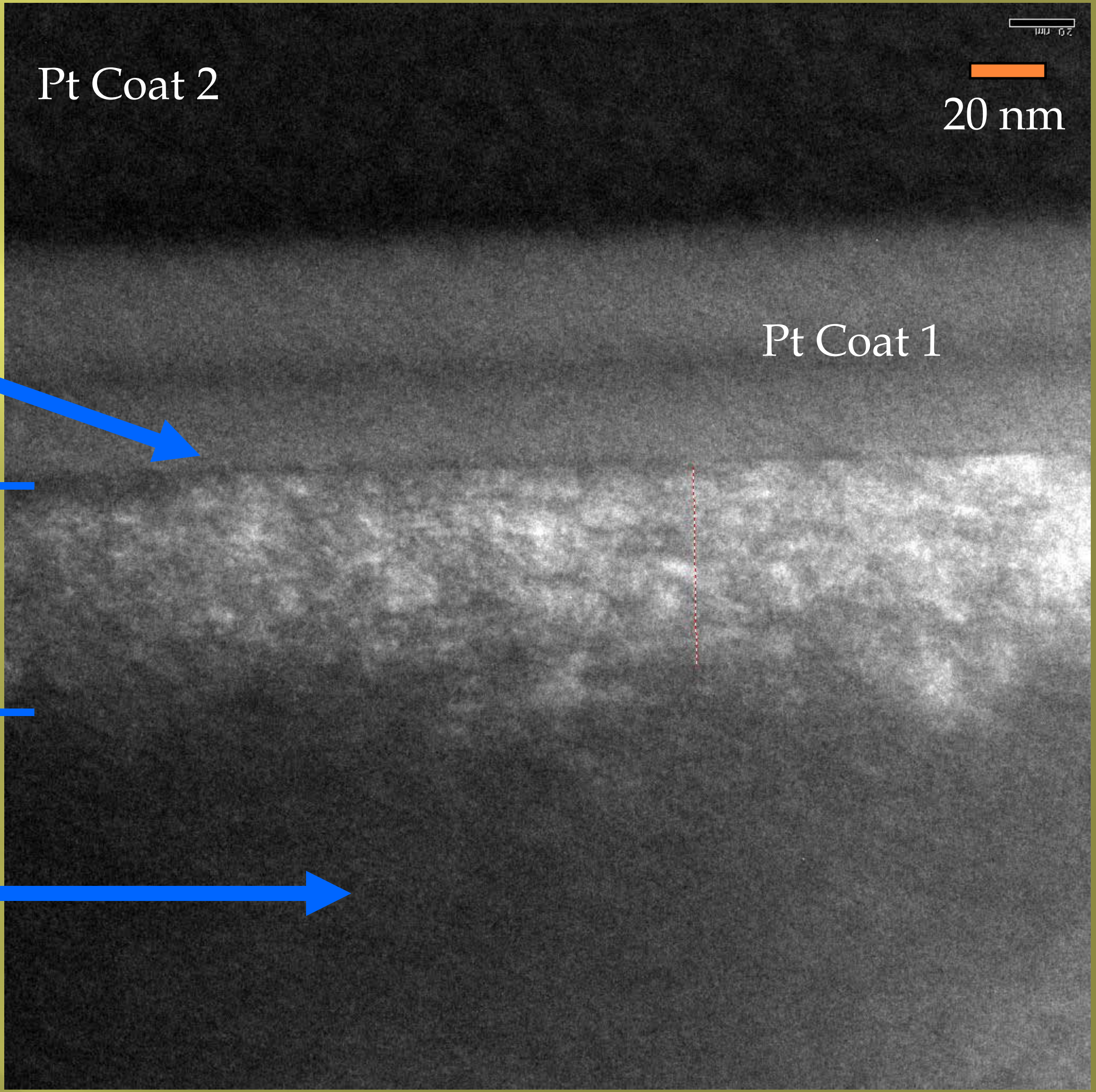
STEM Image of 60208.1 Si B/C Array

Wafer Surface
Native Oxide Layer
SiO_2 = ~ 38 Å

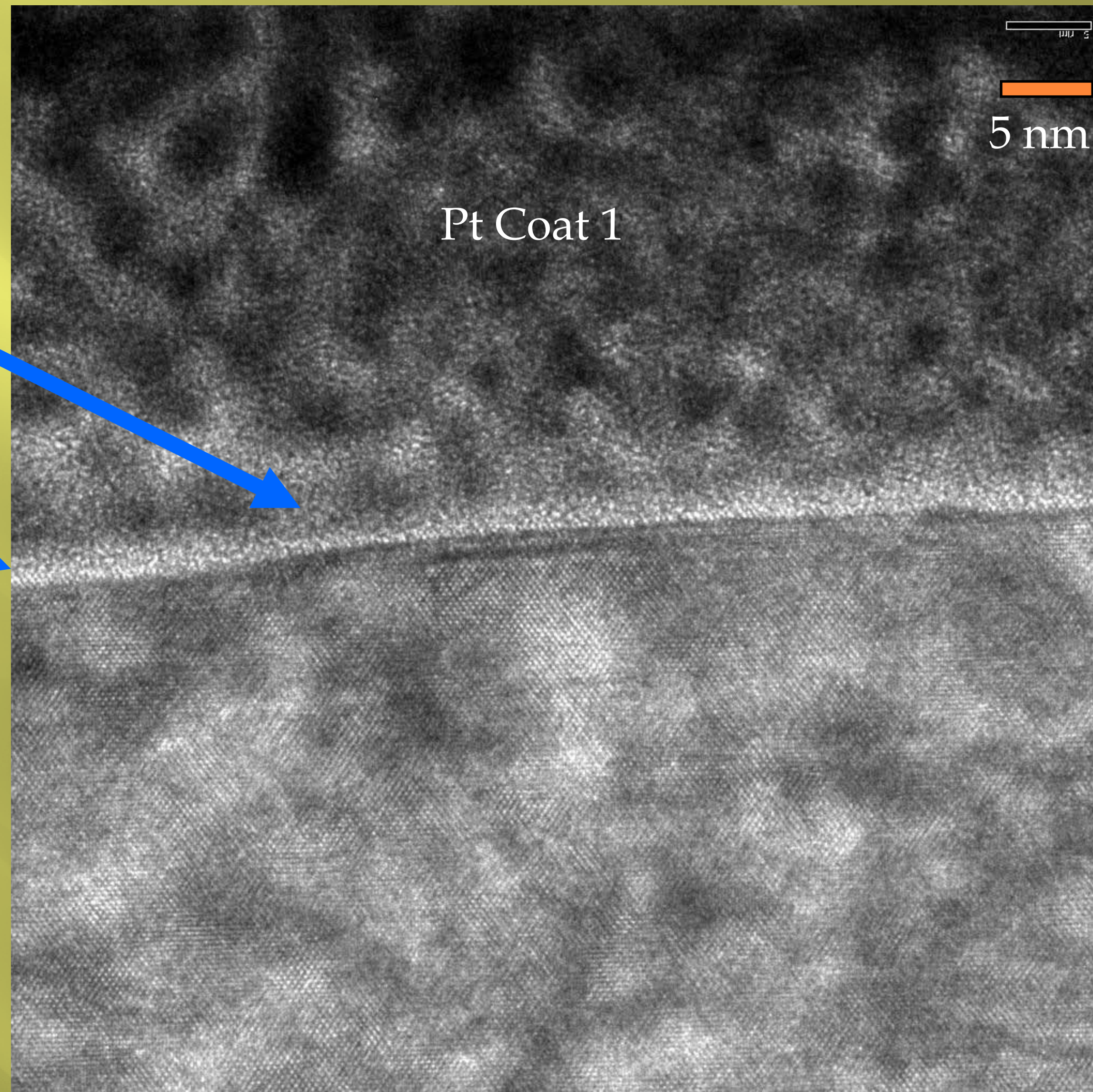
Complex Diffraction Contrast

Radiation Damage Zone
Depth = ~ 610 Å
Range = 60 -70 nm

Crystalline Si Substrate



STEM Image of 60208.1 Si B/C Array

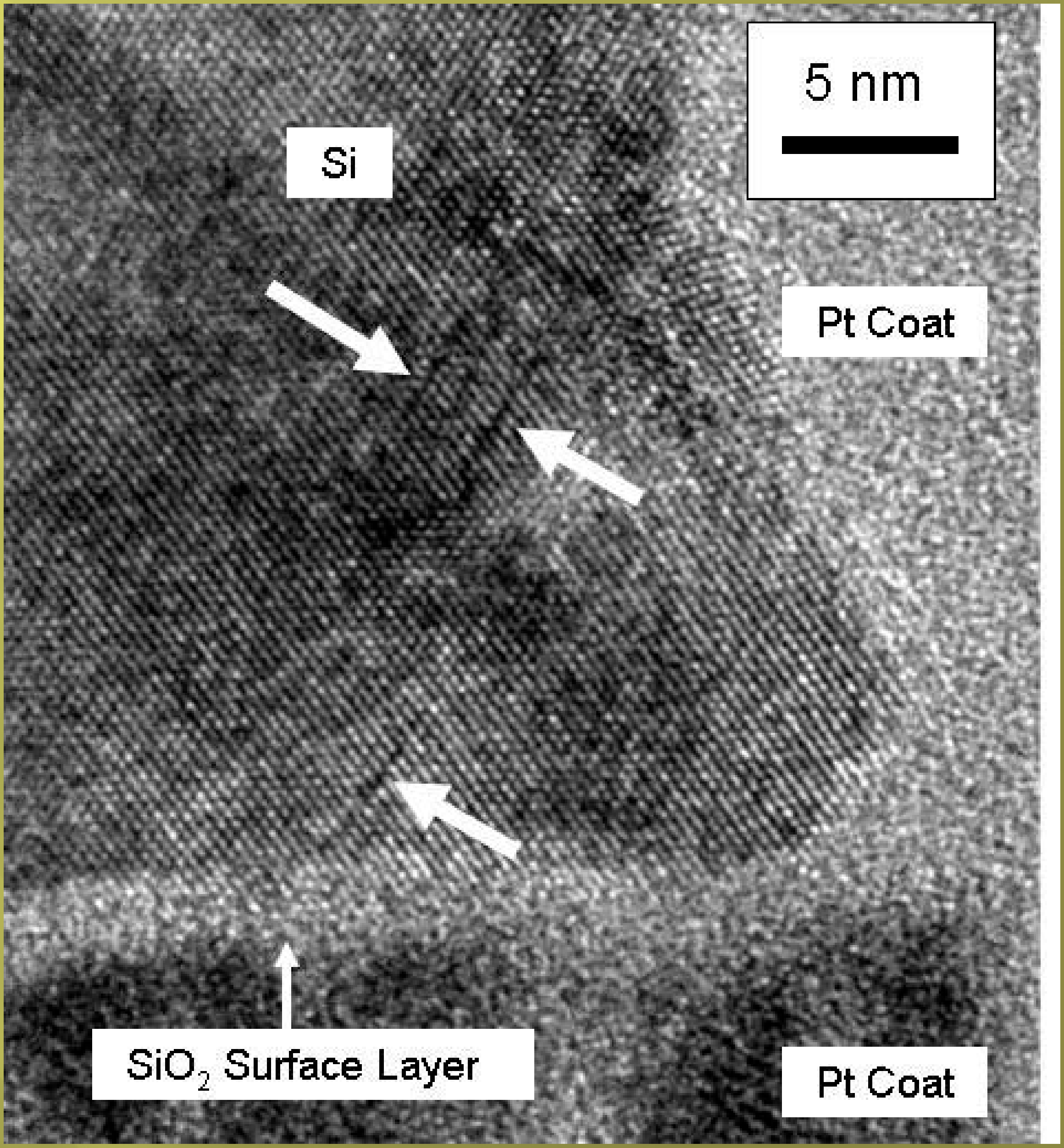


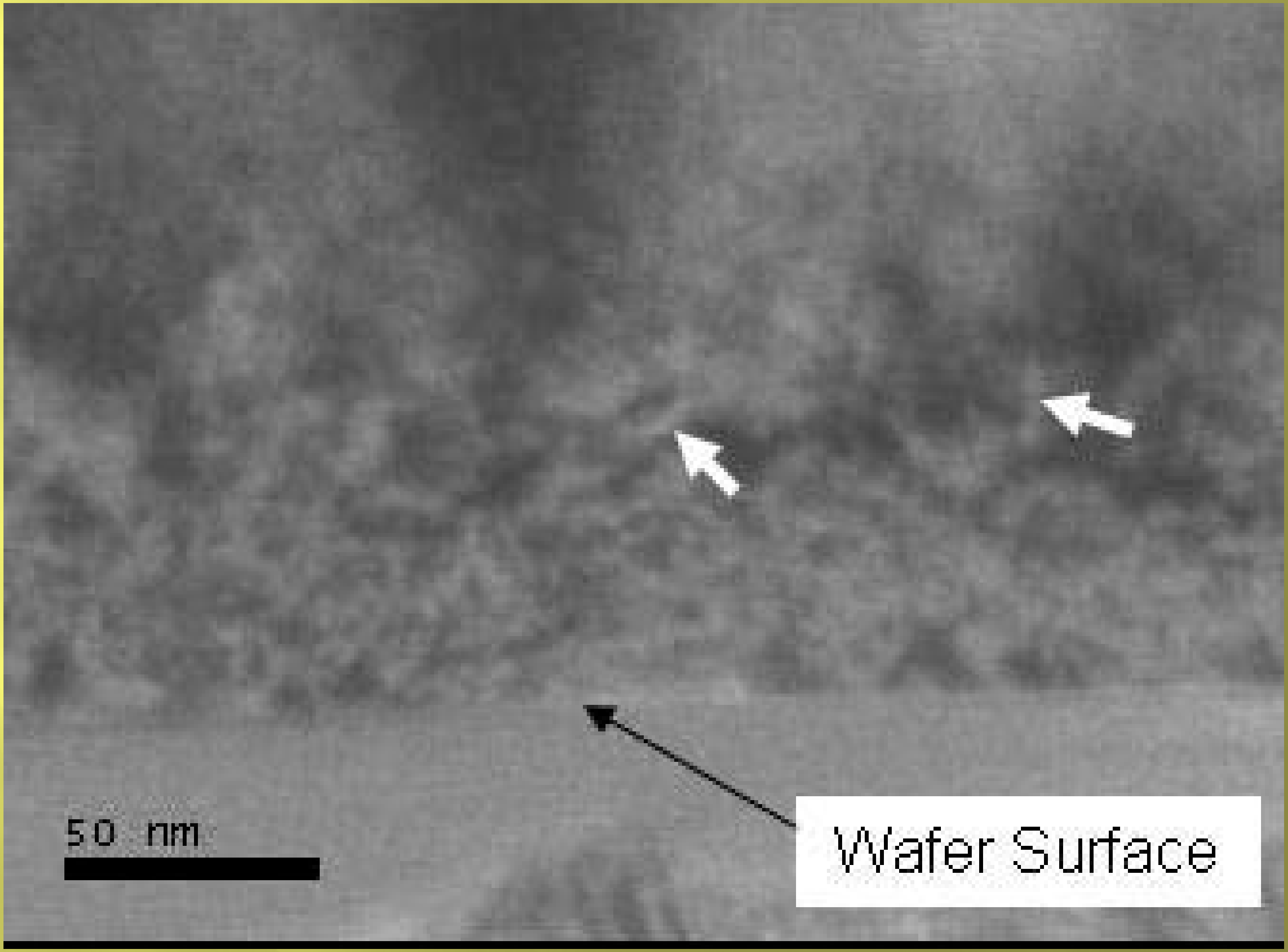
Wafer Surface

Native Oxide Layer
SiO_2 = ~ 38 Å

Radiation Damage Zone

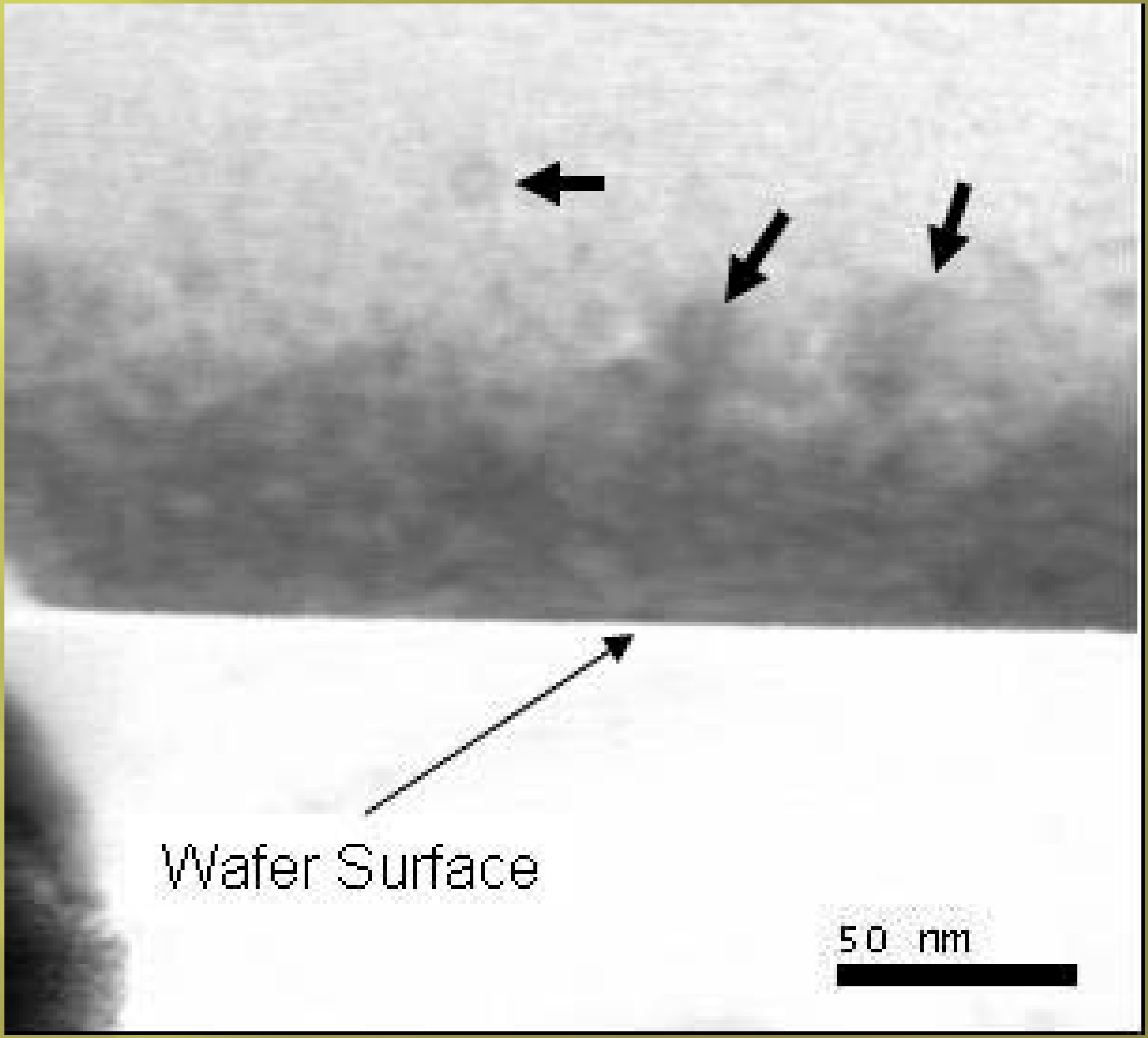
Arrows show possible stacking faults within the strained region directly below the native oxide layer.

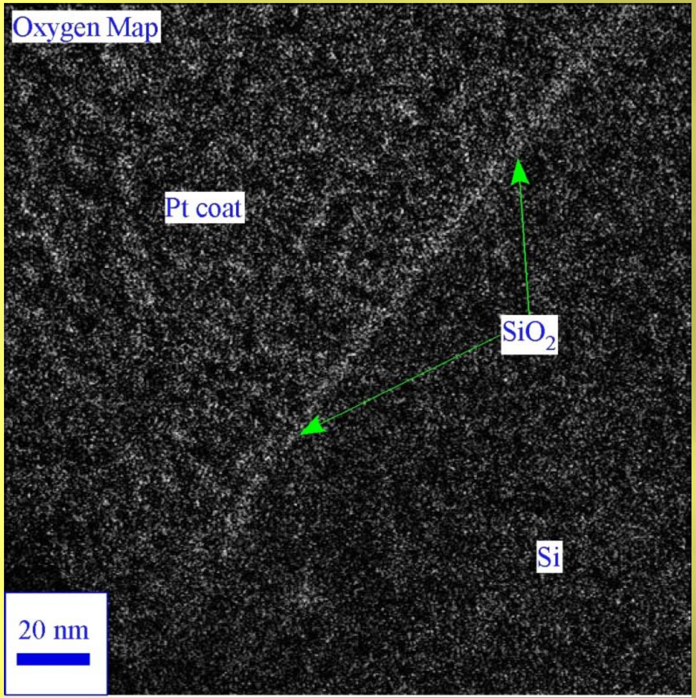




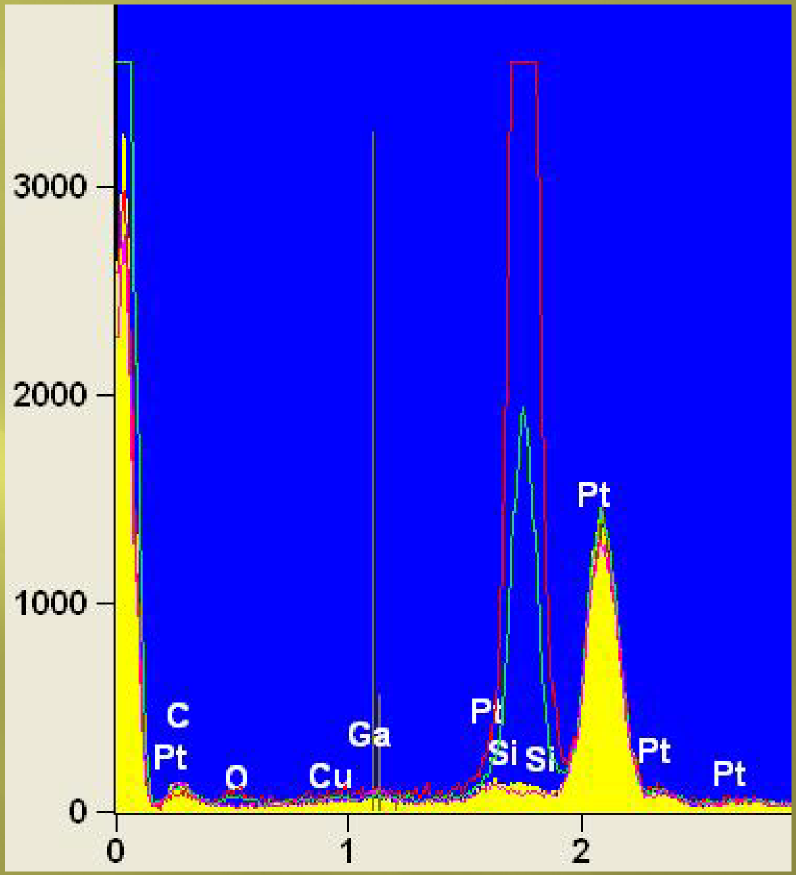
Arrows show possible dislocation line segments within the strained Region.

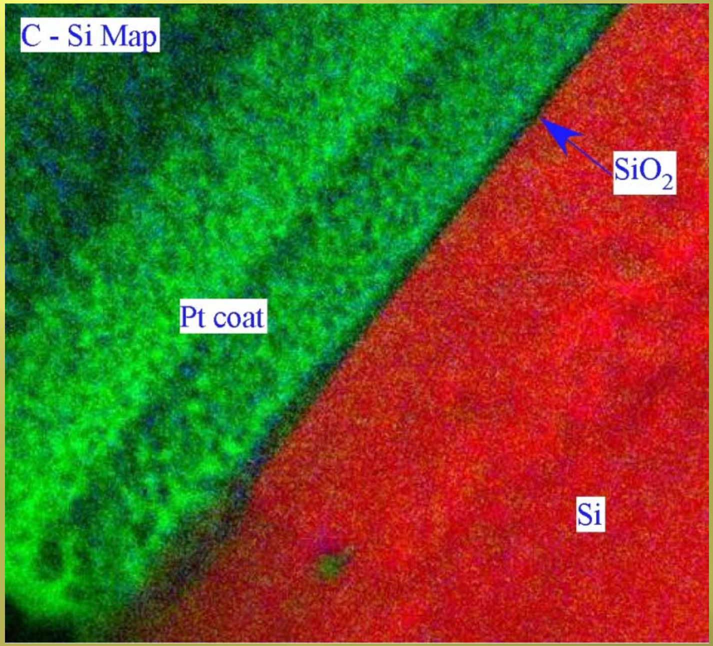
Arrows shows areas of possible dislocation loop defects within the strained region.





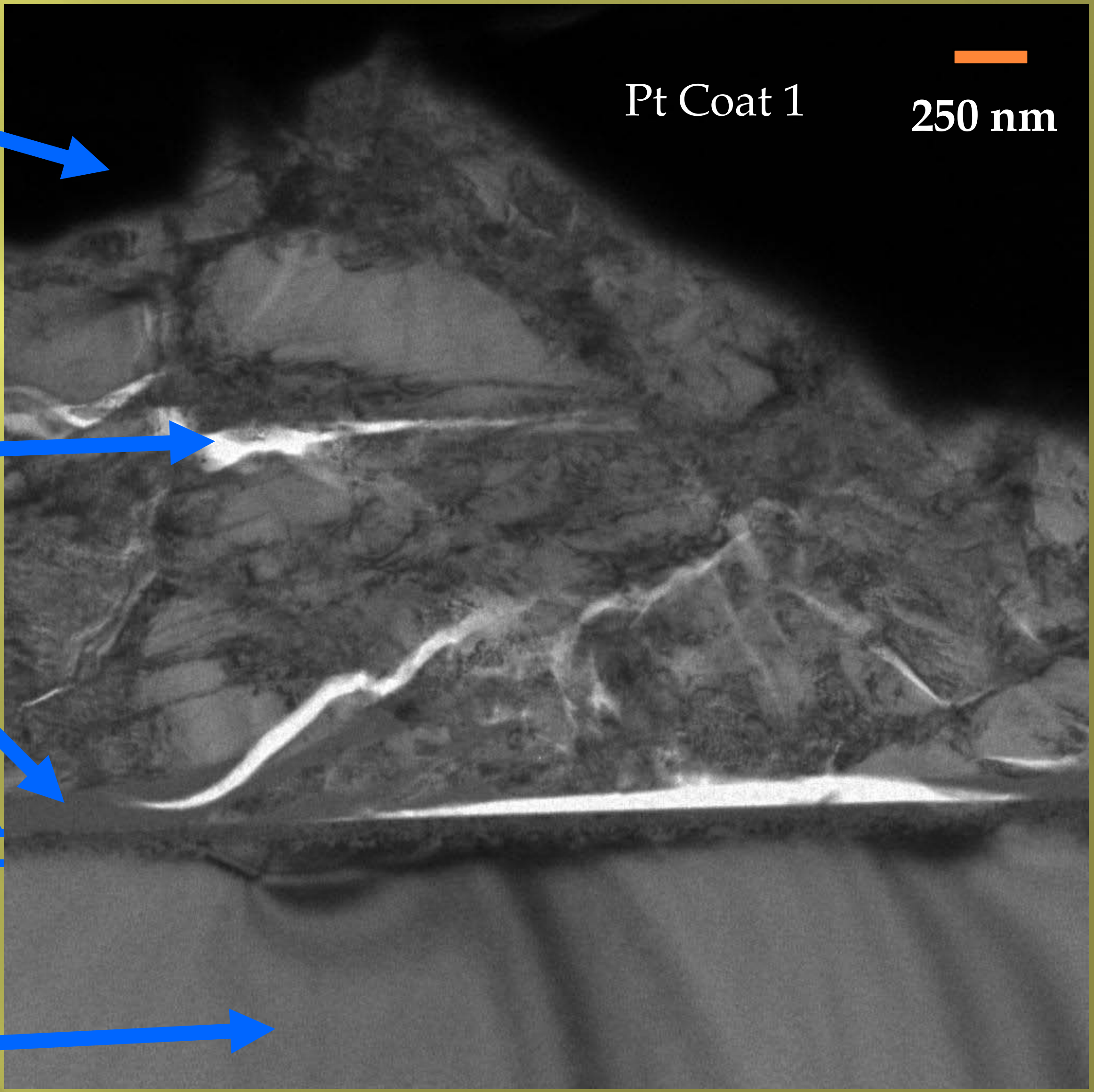
Brown Stain Search with a 1 nm EDS Spot





X-ray EDS spectrum of a 1.0 nm diameter probe transect analysis between the Si substrate and Pt coat. The spectrum clearly shows no change in the C/Pt ratio near the Pt to wafer interface.

STEM Image of 60208.4 Si B/C Array



c-Si Particle on wafer surface (EELS spot verification)

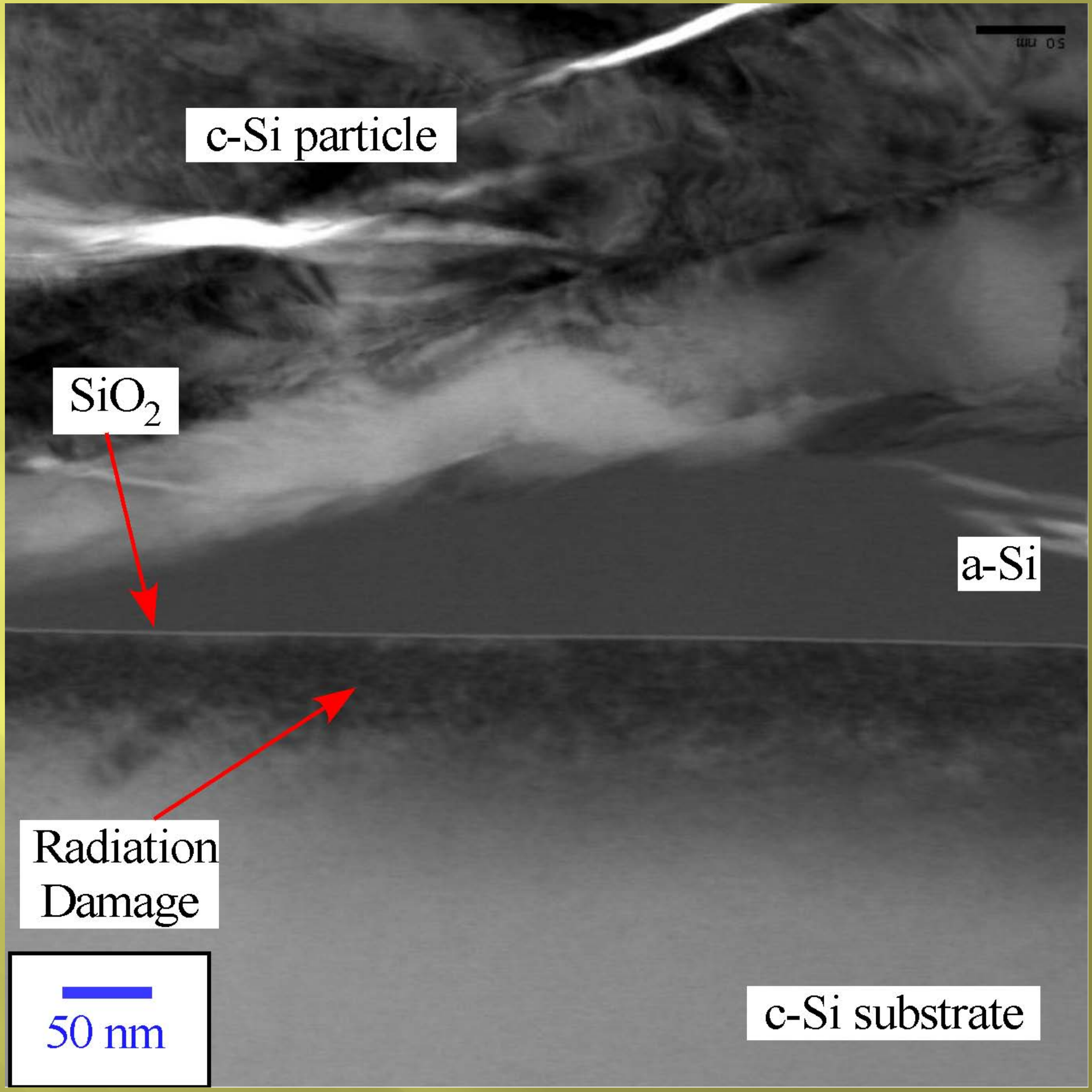
Void Space

a-Si

Wafer Surface

Radiation Damage Zone

c-Si wafer substrate

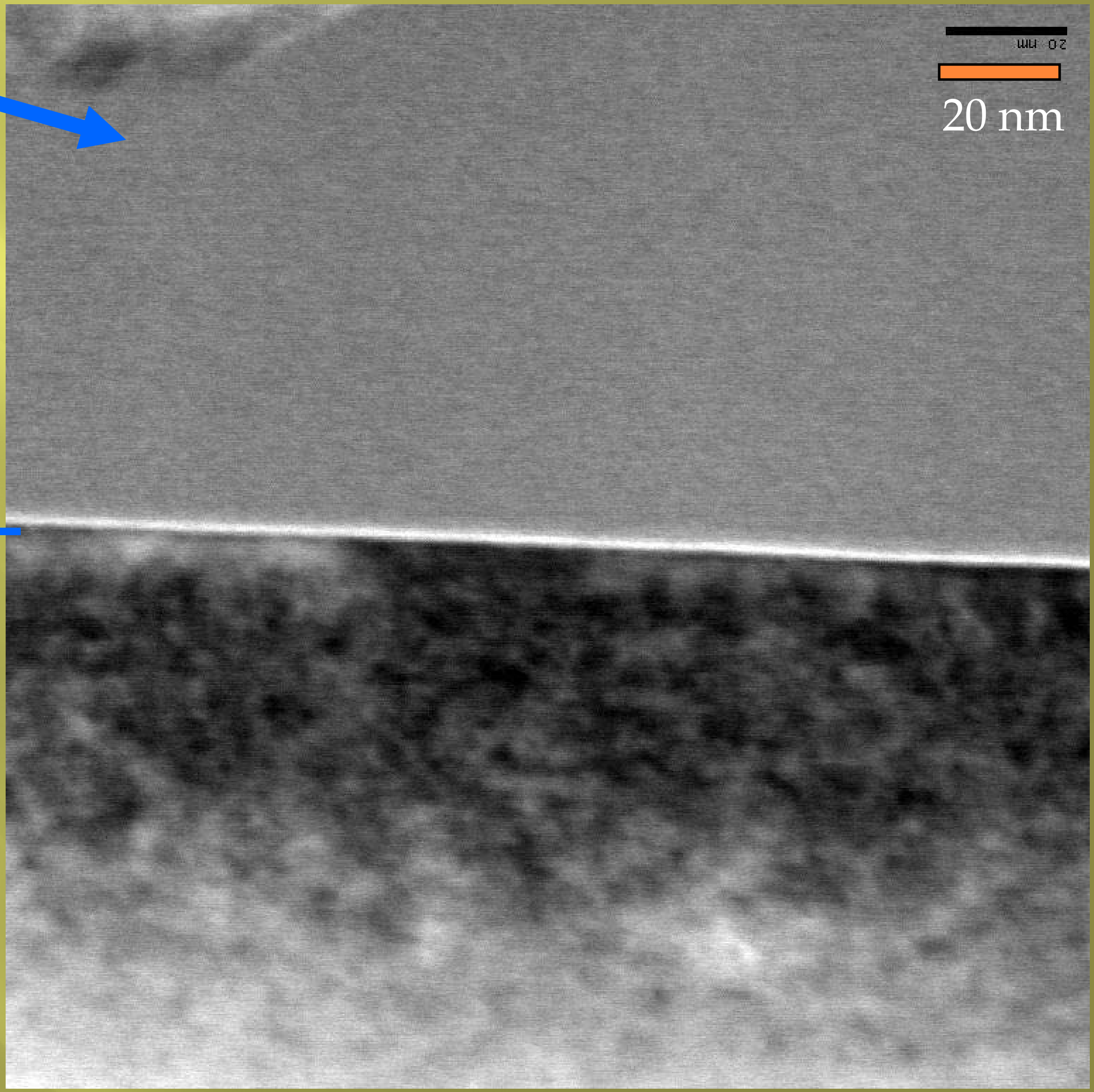

c-Si particle
SiO2
a-Si
Radiation Damage
50 nm
c-Si substrate

STEM Image of 60208.4 Si B/C Array

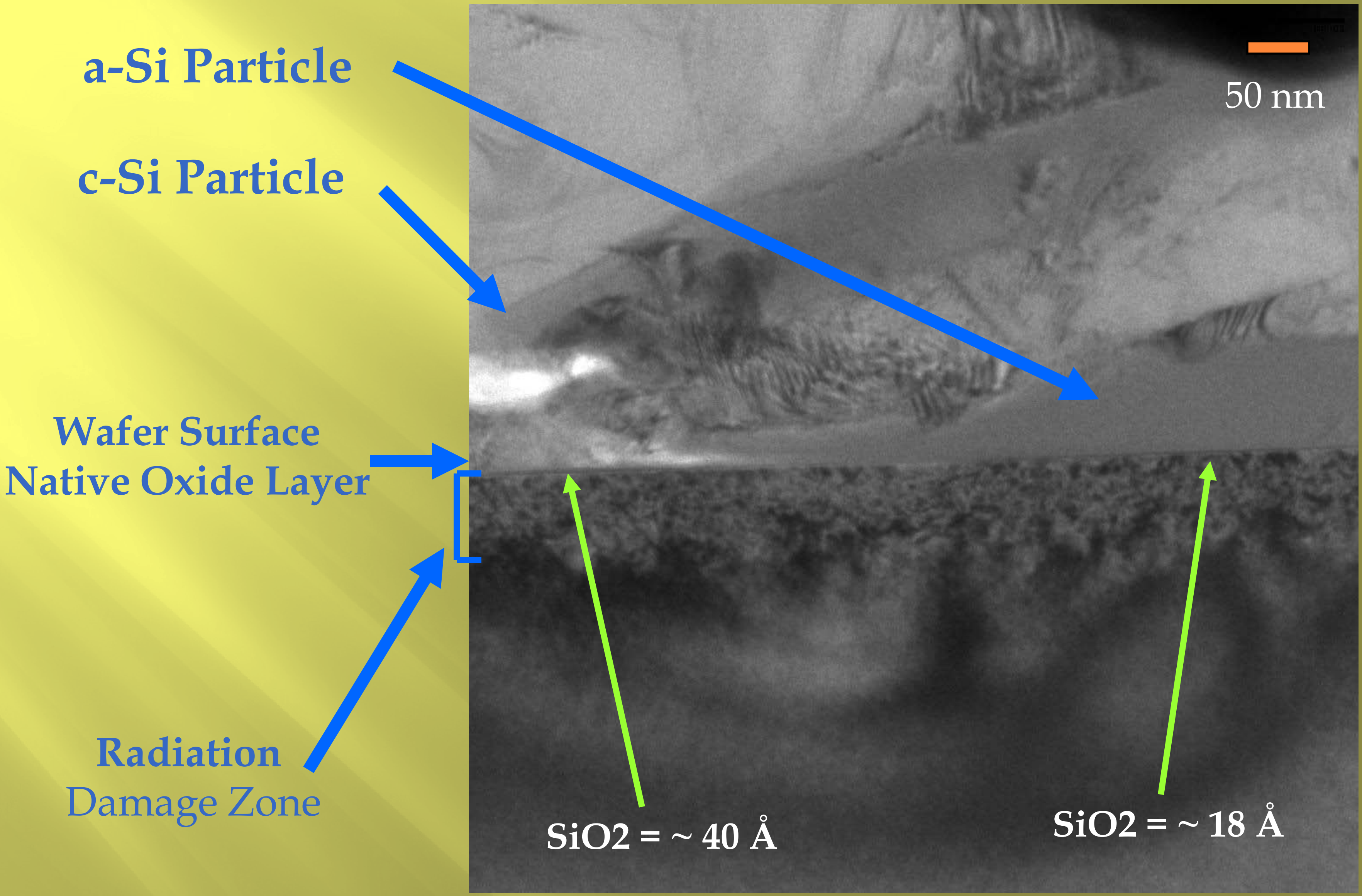
a-Si Particle on wafer surface

Wafer Surface Native Oxide Layer SiO_2 = ~ 18 Å

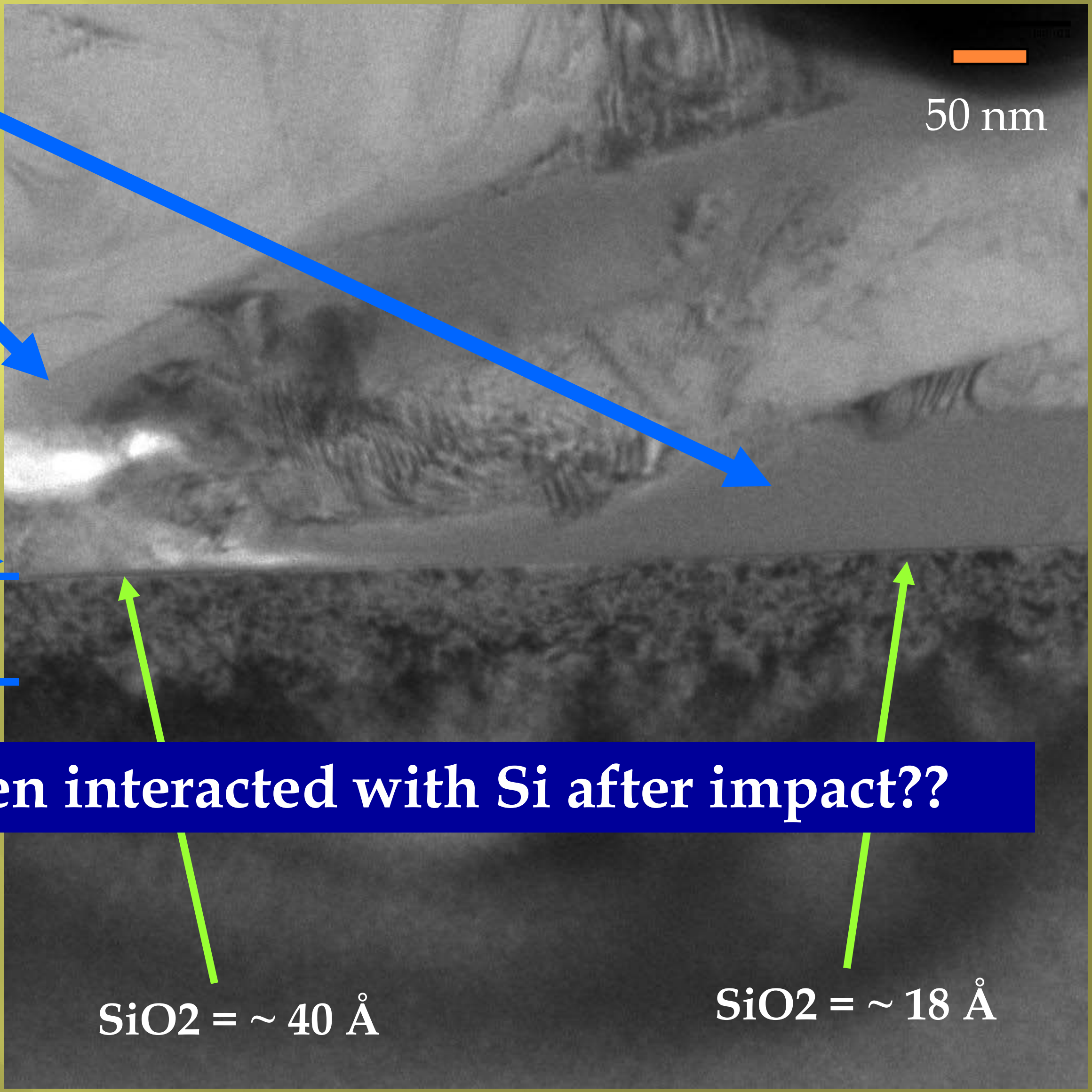
Radiation Damage Zone



STEM Image of 60208.4 Si B/C Array



STEM Image of 60208.4 Si B/C Array



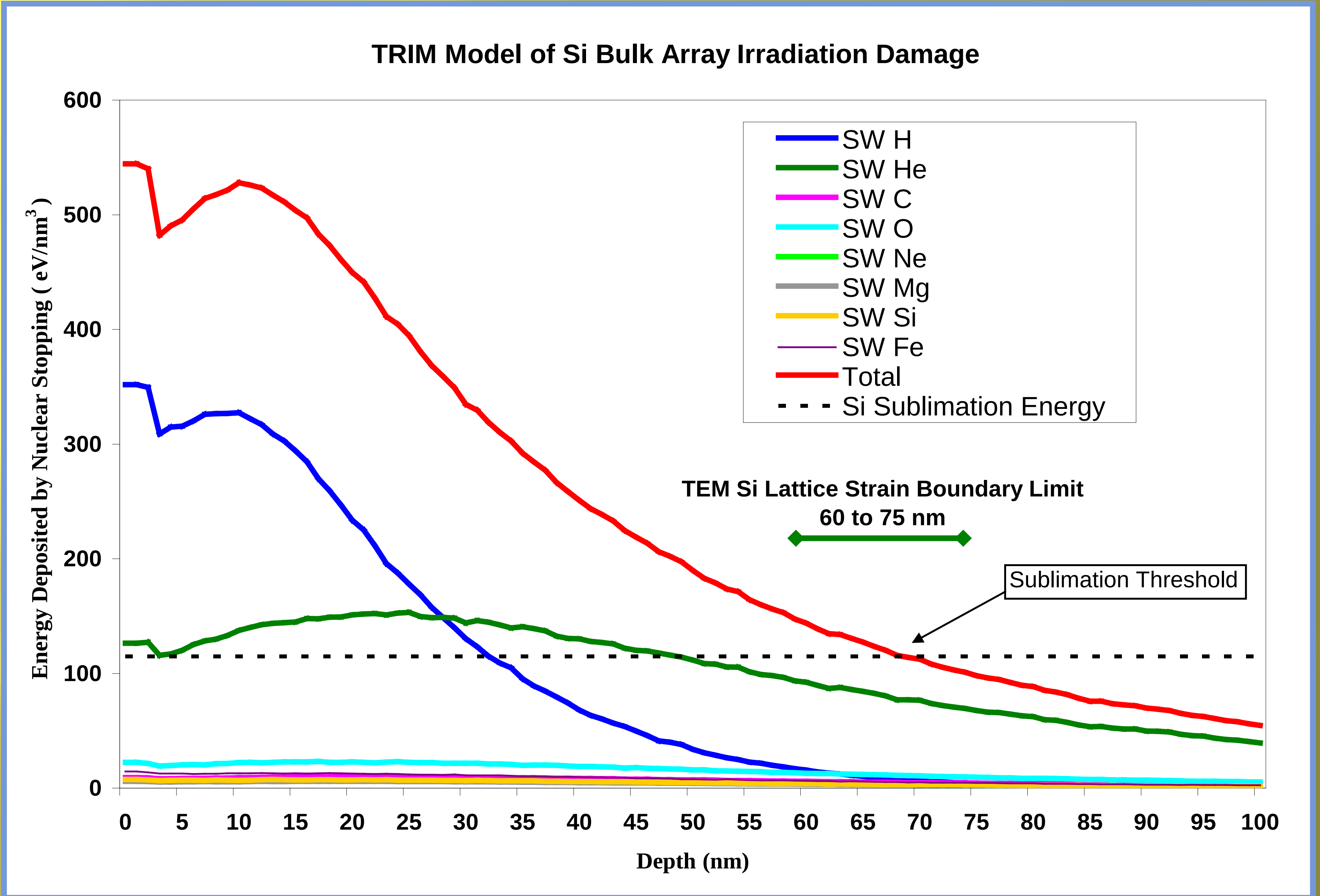
a-Si Particle

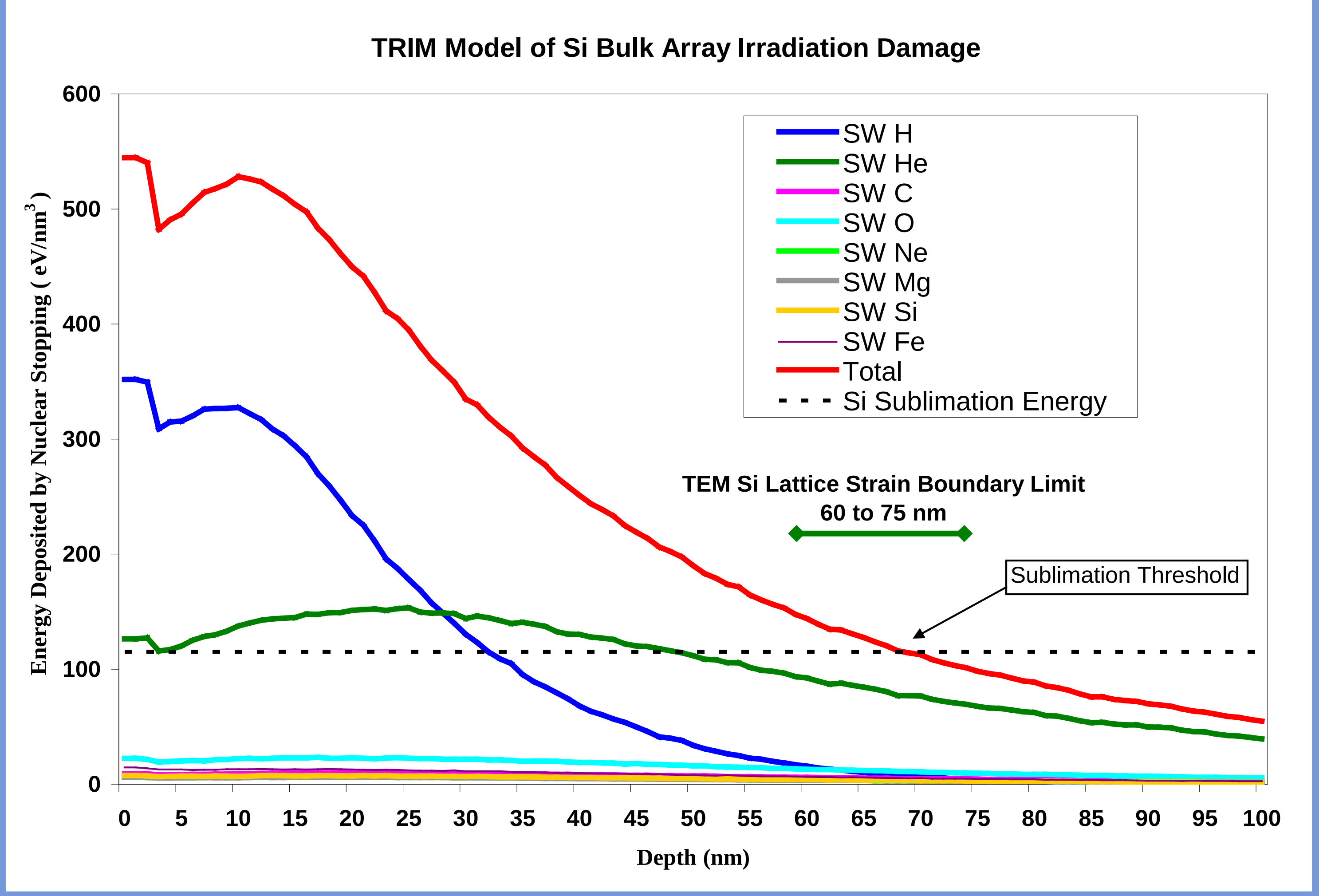
c-Si Particle

Wafer Surface Native Oxide Layer

Radiation Damage Zone

Atmospheric Oxygen interacted with Si after impact??


TRIM Model of Si Bulk Array Irradiation Damage
Energy Deposited by Nuclear Stopping (eV/nm^3)
Depth (nm)
600
500
400
300
200
100
0
0
5
10
15
20
25
30
35
40
45
50
55
60
65
70
75
80
85
90
95
100
SW H
SW He
SW C
SW O
SW Ne
SW Mg
SW Si
SW Fe
Total
Si Sublimation Energy
TEM Si Lattice Strain Boundary Limit
60 to 75 nm
Sublimation Threshold

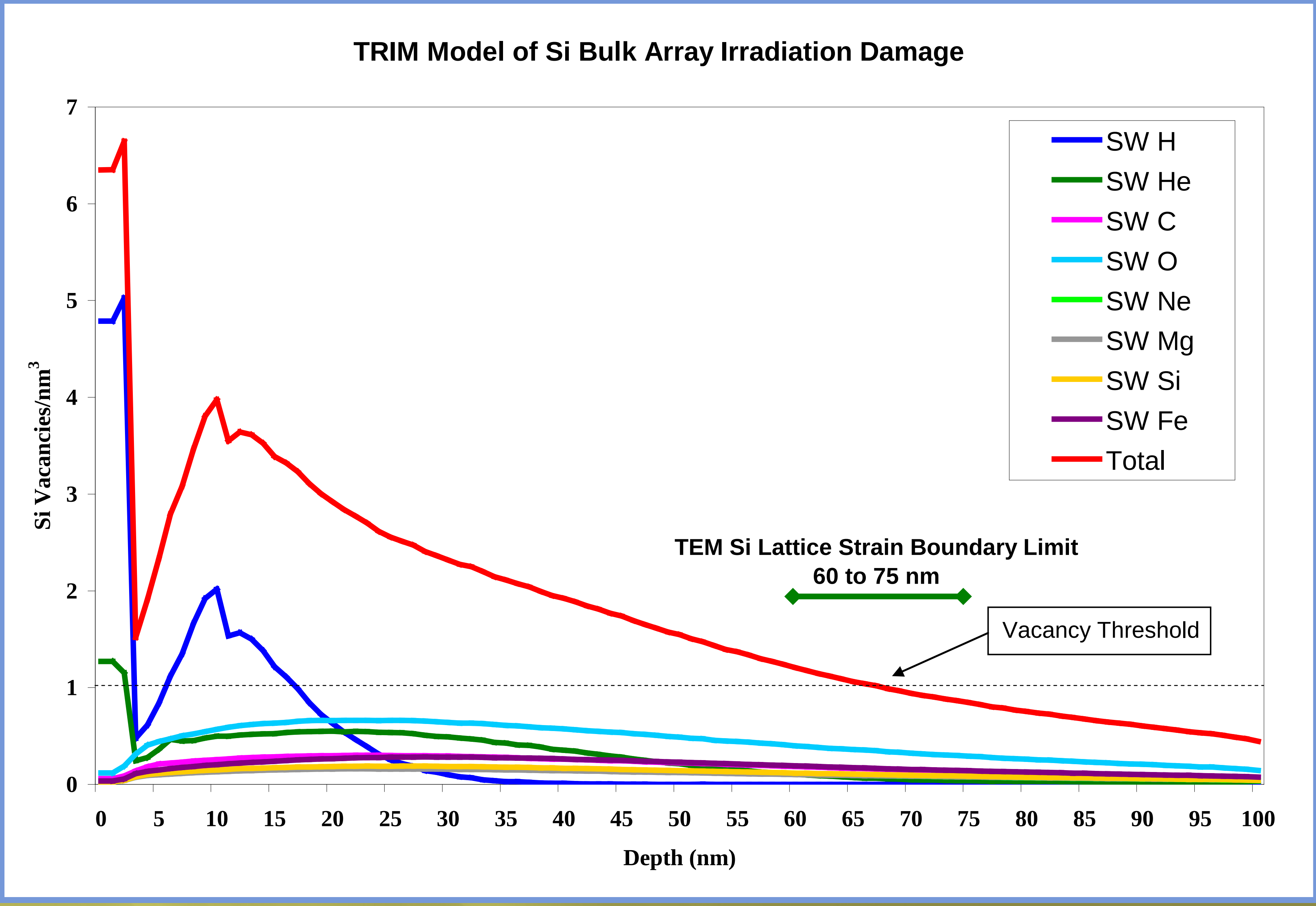


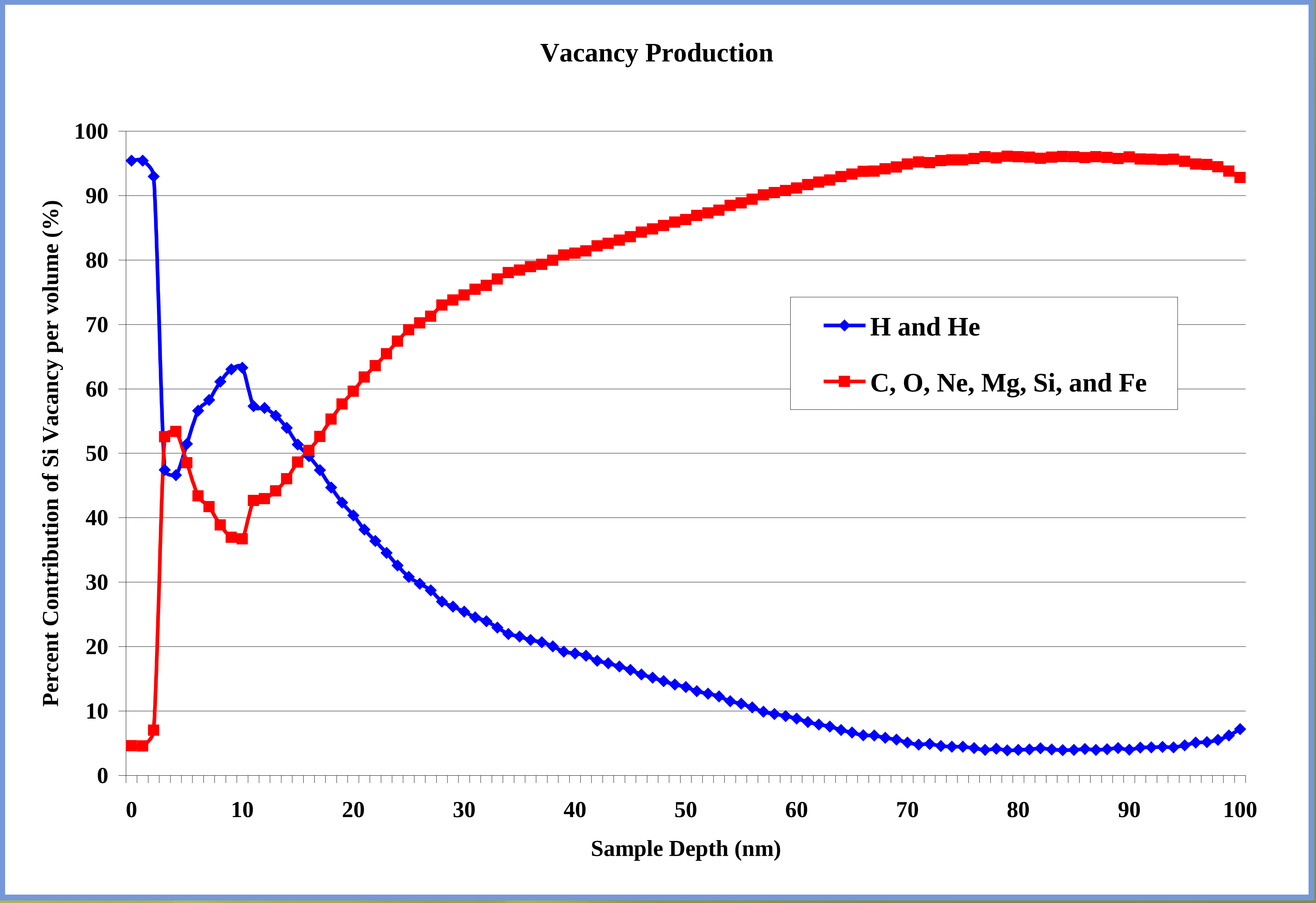
❖Below the threshold for generating a continuous amorphous region at ~ 600 eV/nm^3 (12 eV/atom) [Dennis and Hale 1978; Schreutelkamp et al. 1991]

❖Si vacancy formation at 100 eV/nm^3 (2.0 eV/atom)

❖Si self-interstitials formation at 159 eV/nm^3 (3.18 eV/atom).

[Bracht et al. 1995; Nieminen and Puska 1999; Mozos and Nieminen 1999]


TRIM Model of Si Bulk Array Irradiation Damage
Si Vacancies/nm3
Depth (nm)
0
1
2
3
4
5
6
7
0
5
10
15
20
25
30
35
40
45
50
55
60
65
70
75
80
85
90
95
100
SW H
SW He
SW C
SW O
SW Ne
SW Mg
SW Si
SW Fe
Total
TEM Si Lattice Strain Boundary Limit
60 to 75 nm
Vacancy Threshold


Vacancy Production
Percent Contribution of Si Vacancy per volume (%)
100
90
80
70
60
50
40
30
20
10
0
0
10
20
30
40
50
60
70
80
90
100
Sample Depth (nm)
H and He
C, O, Ne, Mg, Si, and Fe

Conclusions

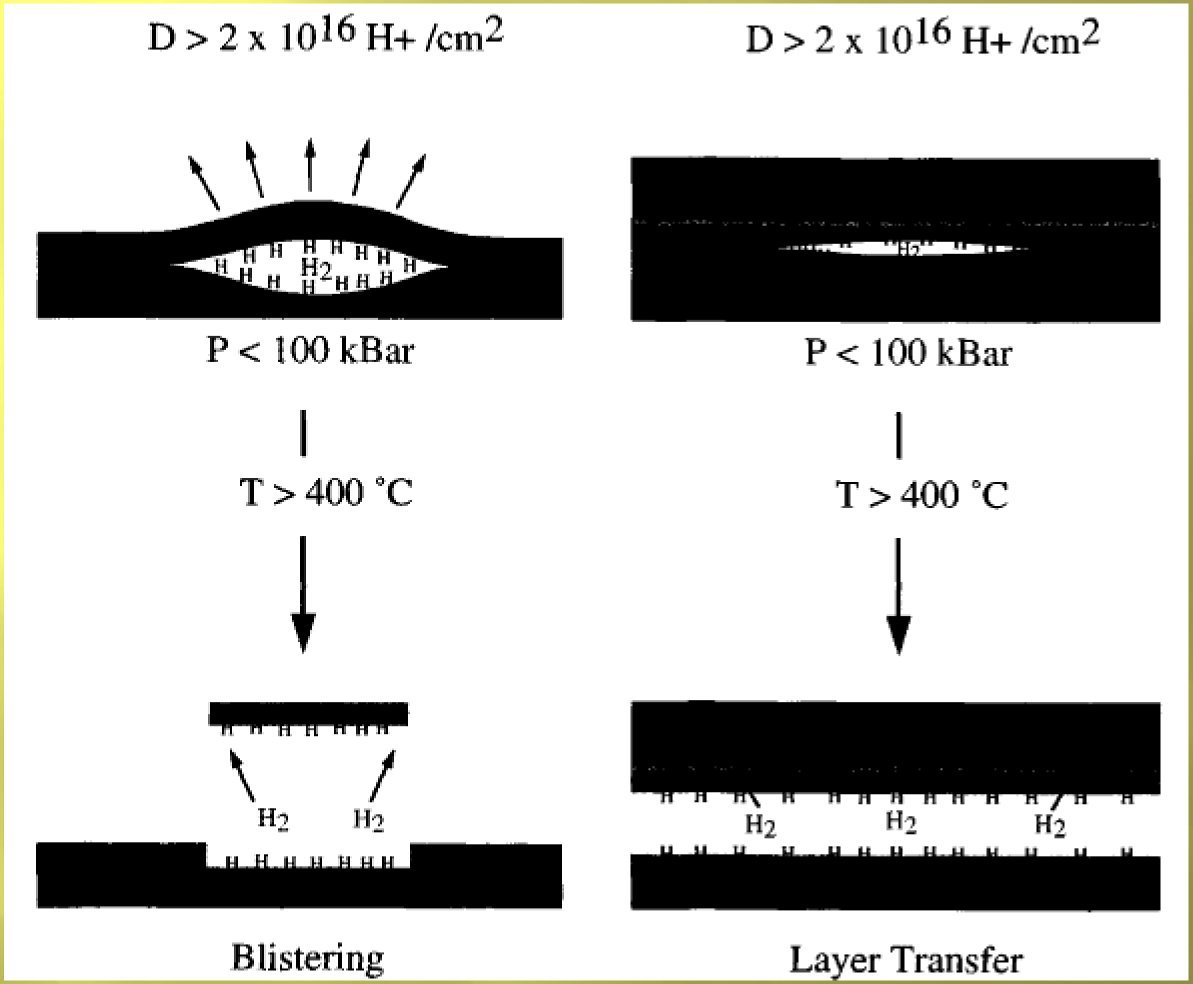
- Majority of the deposited energy was caused by H+ and He+ ion implantation. However, the heavier atomic mass ions (C, O, Ne, Mg, Si and Fe) with much lower fluences could have contributed the majority of the damage at depths greater than 16 nm.

- The fluence threshold required to alter Si materials is below 1.9×10^{16} atoms/cm^2 for solar wind exposure

- Analysis of elemental abundances from Genesis collectors must be aware of changes in the substrate structure due to solar wind irradiation damage that may have occurred throughout the implantation time.

- The results further suggest that surface analysis of any extraterrestrial material must also account for irradiation damage and elemental diffusion when exposed to solar wind.

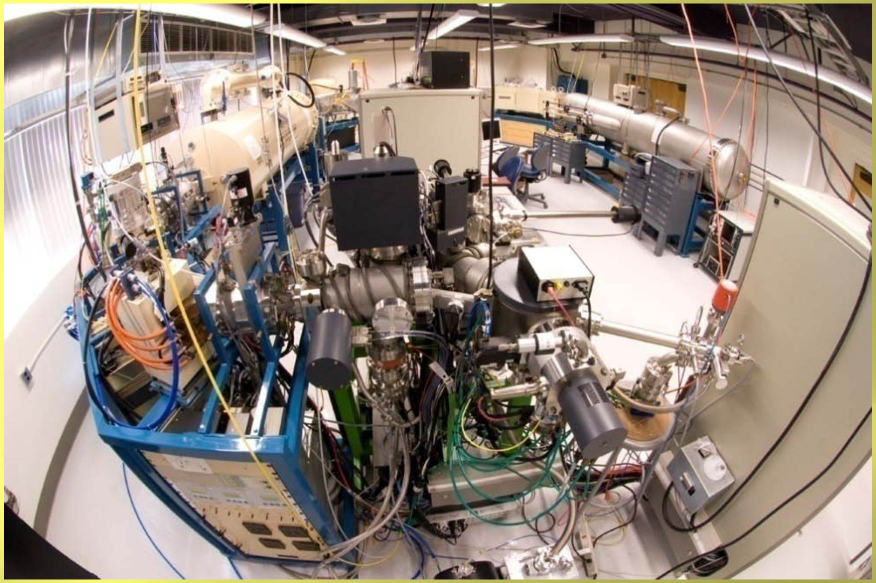
Should we plan a GENSIS II mission with longer exposure times at L1 or on the moon?



10^{17} to 10^{20} H^+ atoms/cm^2 may cause exfoliation in pure crystalline silicon

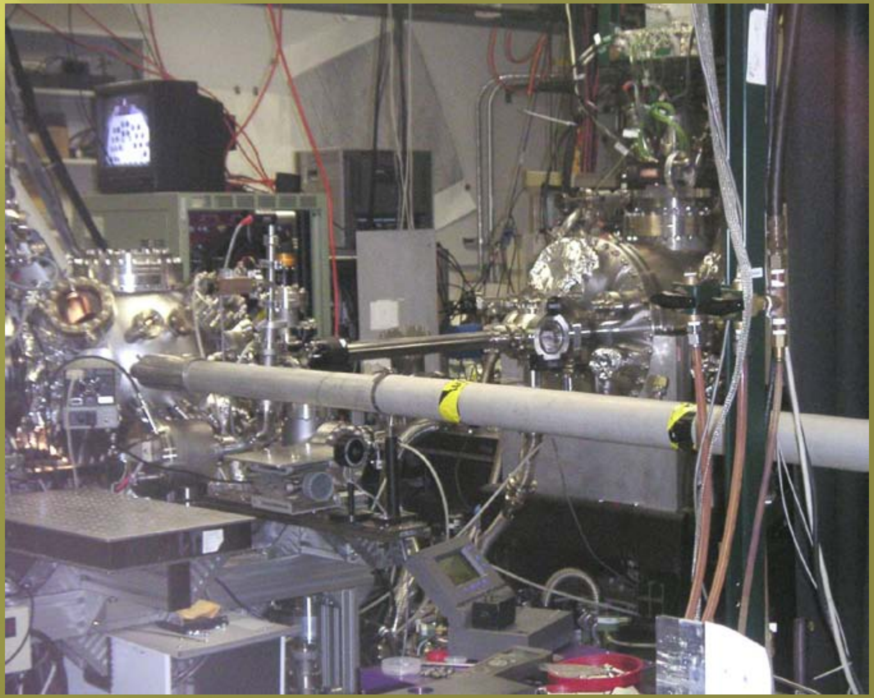
Weldon and Chabal (1999) **Hydrogen-induced exfoliation of c-Si,** in: R. Hull (Ed.), Properties of Crystalline Silicon, INSPEC, London, p. 942.

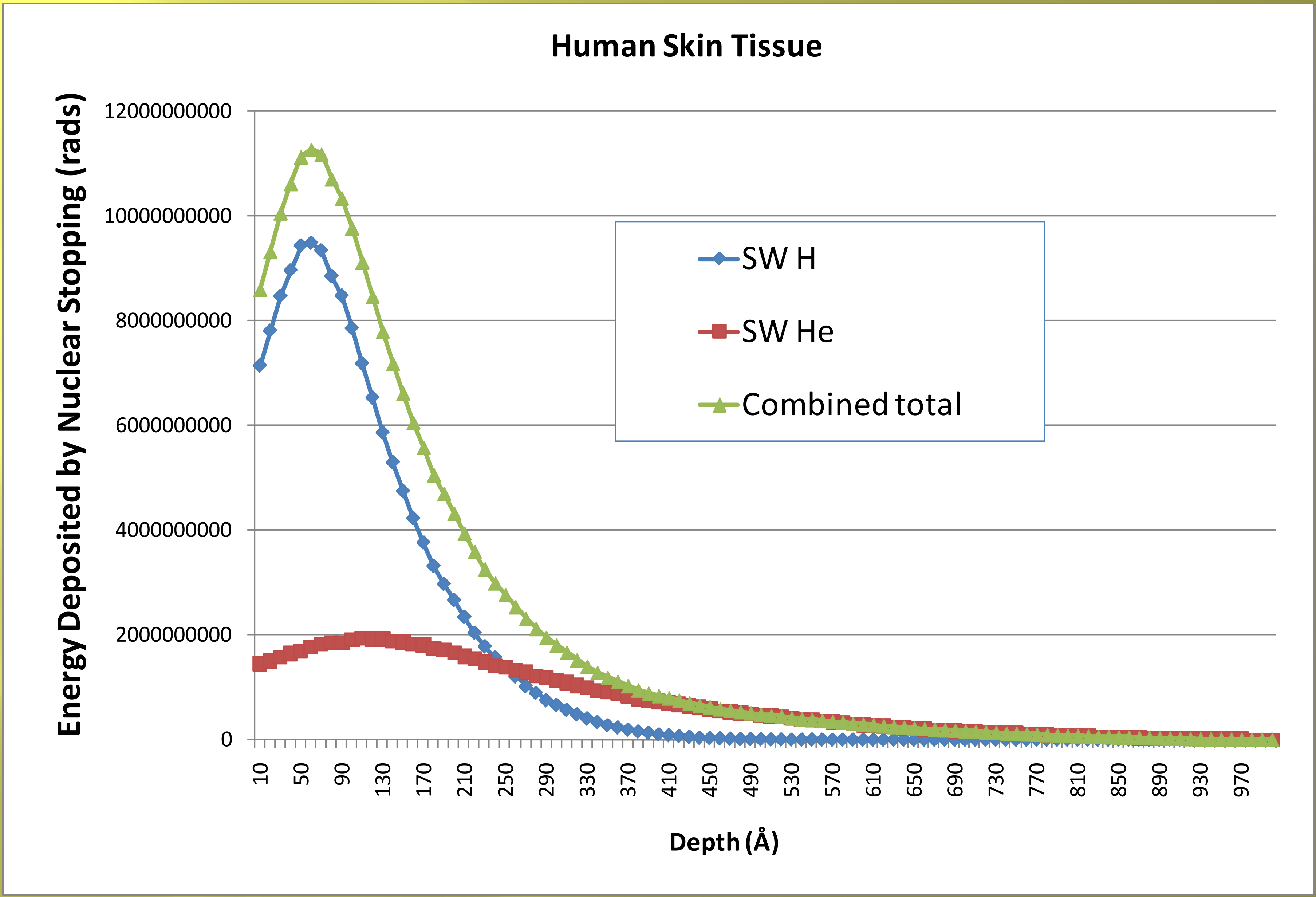
GENESIS's need for Advanced Surface Analysis Techniques



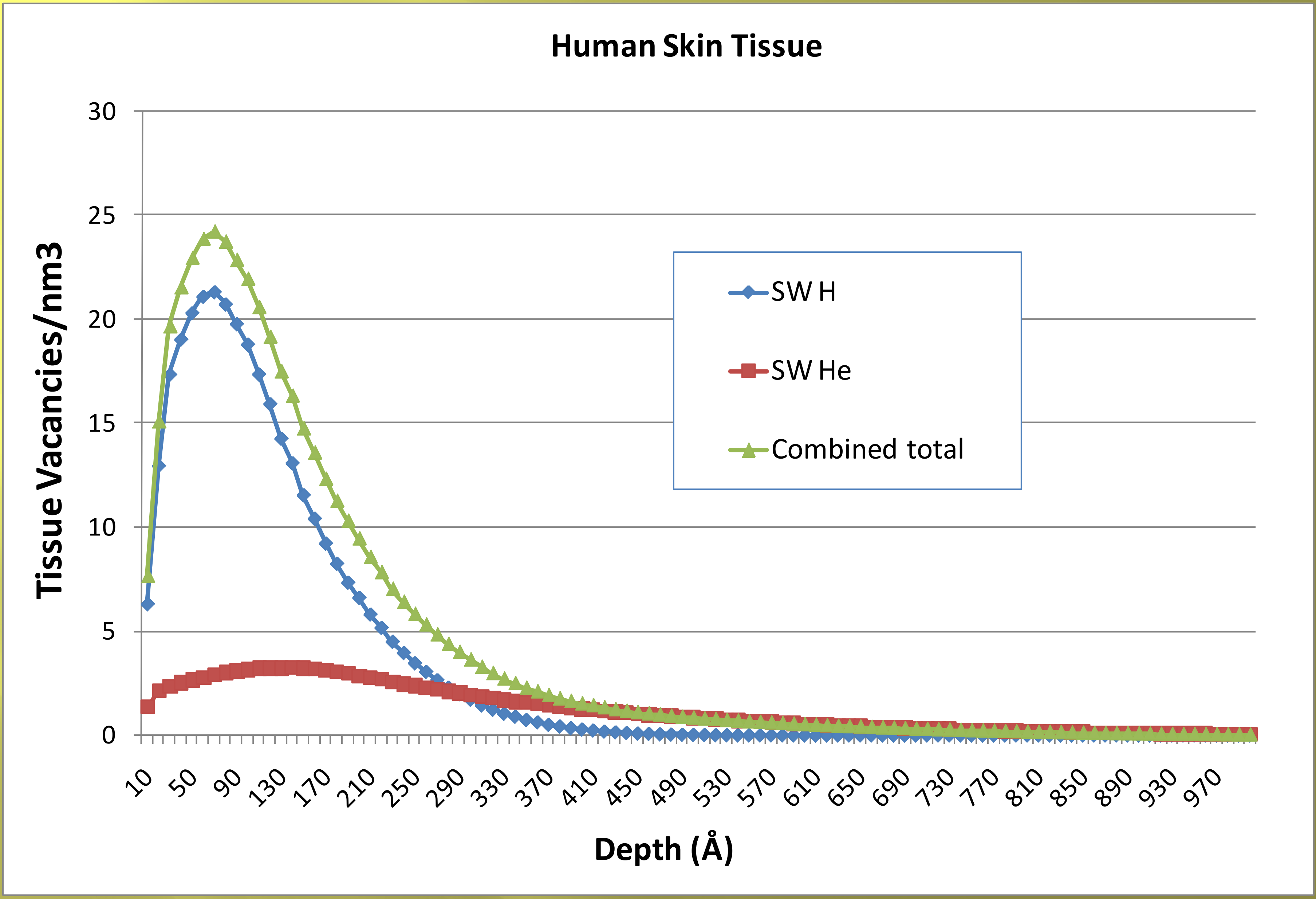
MegaSIMS at UCLA

SARISA (Surface Analysis by Resonance Ionization of Sputtered Atoms) at Argonne National Lab

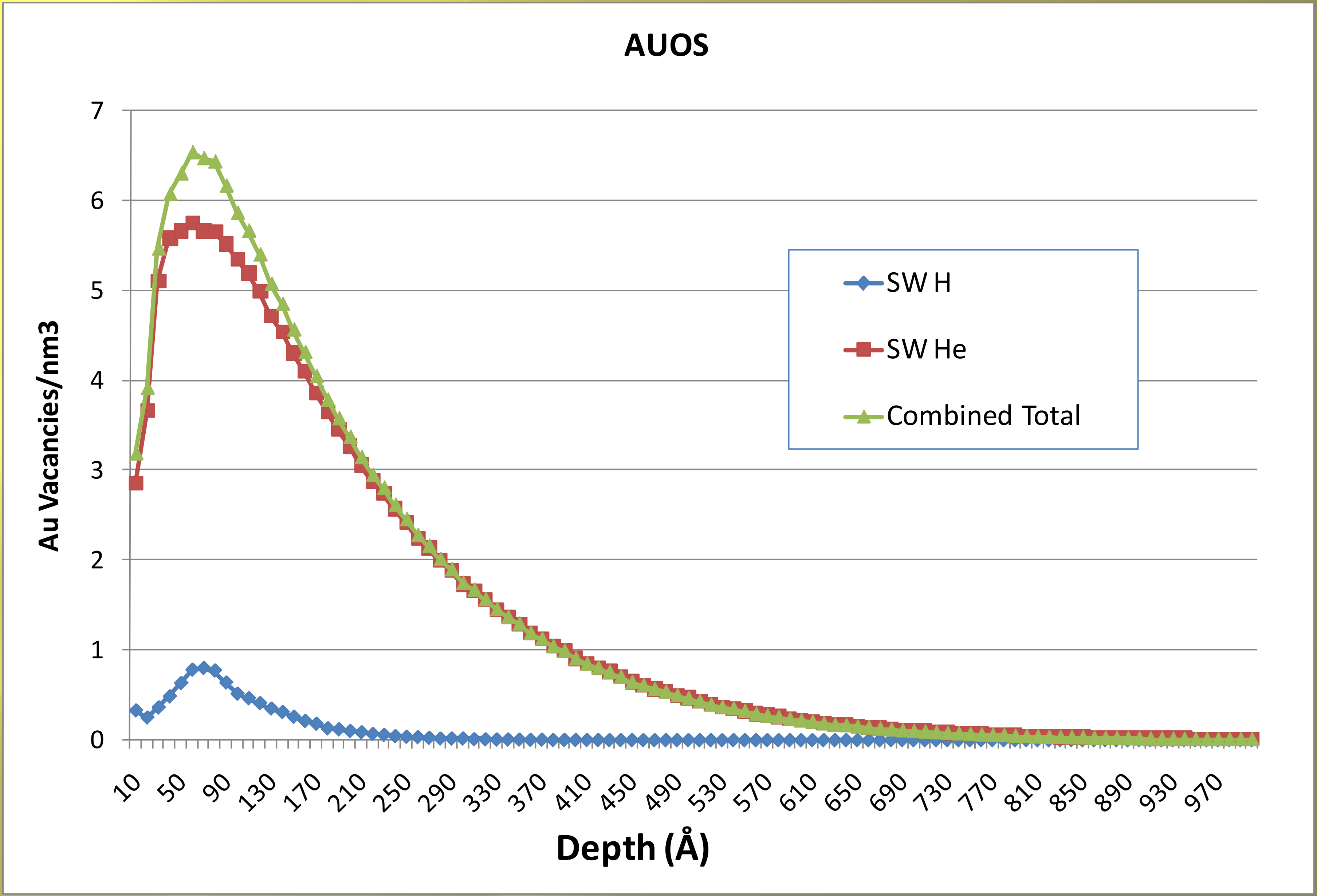




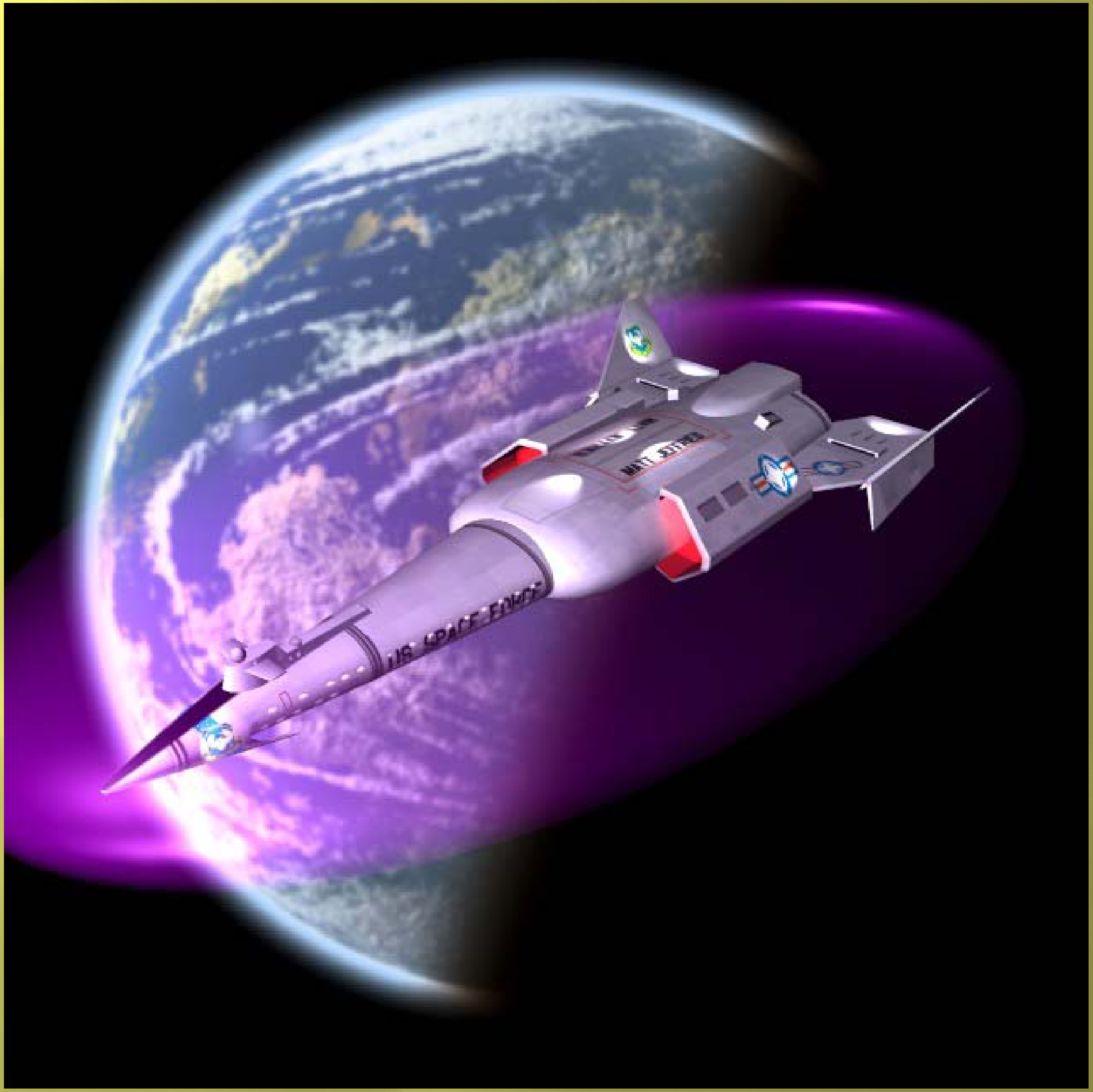
Human Skin Tissue
Energy Deposited by Nuclear Stopping (rads)
12000000000
10000000000
8000000000
6000000000
4000000000
2000000000
0
10
50
90
130
170
210
250
290
330
370
410
450
490
530
570
610
650
690
730
770
810
850
890
930
970
Depth (Å)
SW H
SW He
Combined total

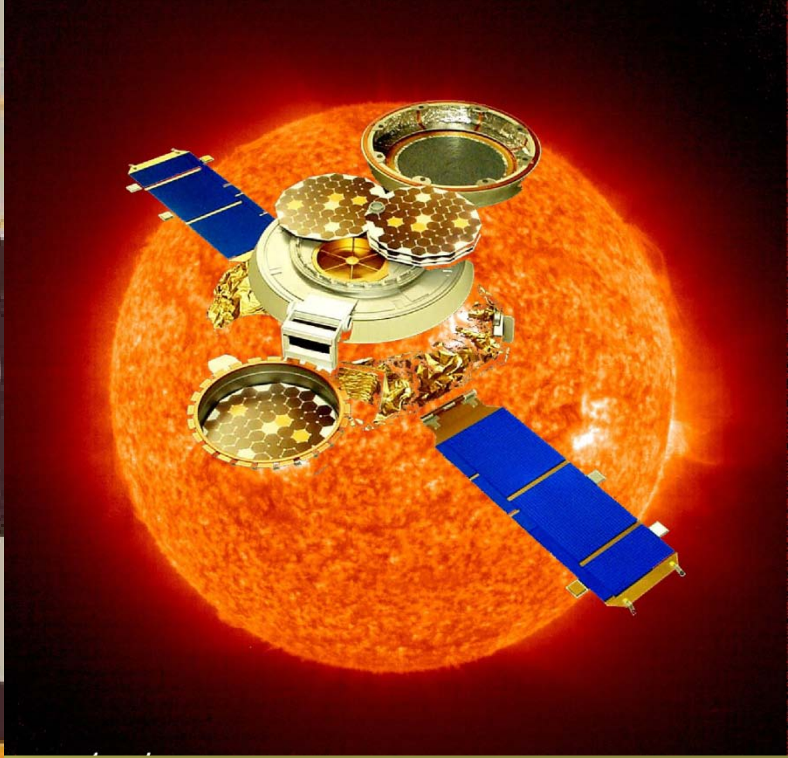


Human Skin Tissue
Tissue Vacancies/nm3
30
25
20
15
10
5
0
SW H
SW He
Combined total
10
50
90
130
170
210
250
290
330
370
410
450
490
530
570
610
650
690
730
770
810
850
890
930
970
Depth (Å)


AUOS
Au Vacancies/nm3
Depth (Å)
0
1
2
3
4
5
6
7
10
50
90
130
170
210
250
290
330
370
410
450
490
530
570
610
650
690
730
770
810
850
890
930
970
SW H
SW He
Combined Total

The Need for Advancements in Radiation Protection





Questions

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BEAM INTERACTIONS WITH MATERIALS AND ATOMS

Genesis capturing the sun: Solar wind irradiation at Lagrange 1

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Silicon defects

A B S T R A C T

Genesis, a member of NASAs Discovery Mission program, is the world's first sample return mission since the Apollo program to bring home solar matter in ultra-pure materials. Outside the protection of Earth's magnetosphere at the Earth–Sun Lagrange 1 point, the deployed sample collectors were directly exposed to solar wind irradiation. The natural process of solar wind ion implantation into a highly pure silicon (Si) bulk composition array collector has been measured by spectroscopic ellipsometry and scanning transmission electron microscopy (STEM). Ellipsometry results show that bulk solar wind ions composed of approximately 95% H^+, 4% He^+ and <1% other elements physically altered the first 59–63 nm of crystalline silicon substrate during 852.8 days of solar exposure. STEM analysis confirms that the solar accelerated ions caused significant strain and visible structural defects to the silicon structure forming a 60–75 nm thick irradiation damage region directly below the surface SiO_2 native oxide layer. Monte Carlo simulations of solar wind H, He, C, O, Ne, Mg, Si and Fe ion collisions in the Si collector with fluences calculated from the Genesis and ACE spacecrafts were used to estimate the energy deposited and Si vacancies produced by nuclear stopping in a flight-like Si bulk array collector. The coupled deposited energy model with the flown Genesis Si in situ measurements provides new insight into the basic principles of solar wind diffusion and space weathering of materials outside Earth's magnetosphere.