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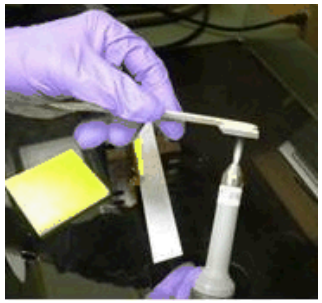

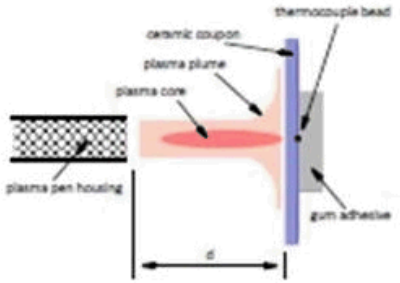

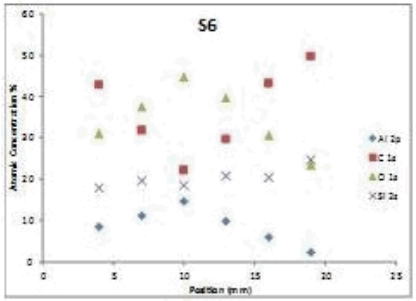

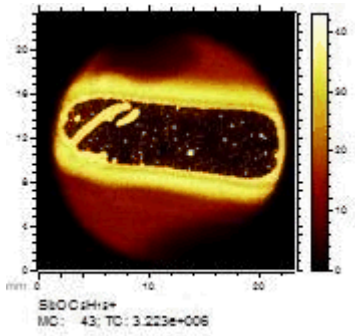

Plasma Pen Characterization

For Ceramic Substrate, Part 1

Kim Archuleta, James Ohlhausen, Michael Brumbach, Carlton Brooks, Anne Grillet, Richard Givler and Allen Roach

U.S. DEPARTMENT OF ENERGY | NNSA National Nuclear Security Administration

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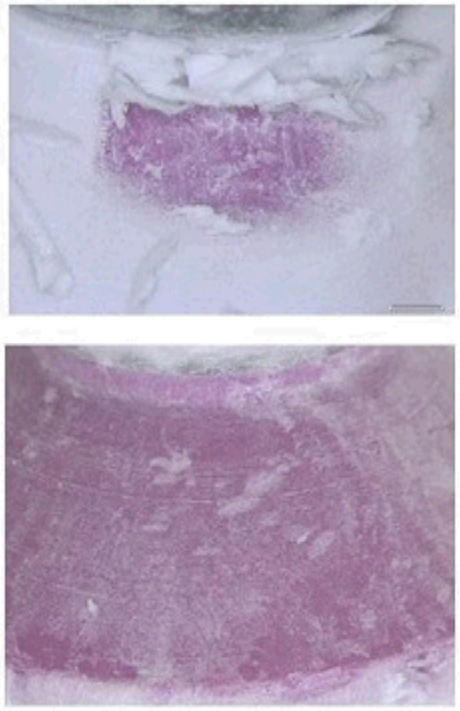
Background: De-bonding of encapsulant on ceramic

- Physical and electrical stresses cause delamination of encapsulant.
- Review of process indicates less than optimal cleaning and handling

94% alumina ceramic historically has had instances of poor epoxy encapsulant bond quality.

Encapsulant detaches when stressed sufficiently.

Background (Cont.)



- Adding detergent cleaning step resulted in increased adherence of encapsulant to ceramic.
- After stressing, encapsulant remains well attached.
- Increasing cleaning in areas of highest stress may further strengthen the encapsulant bond.

Postulate: Cleaning for improved bonding

- Clean surface vital for welding, brazing, coating and encapsulation
- Treatment required immediately before bond process for optimal result
- Conventional means of cleaning may not be applicable
    - Part cannot be submersed
    - Areas of part cannot be exposed to some cleaning agents

Postulate: Cleaning for improved bonding (cont.)

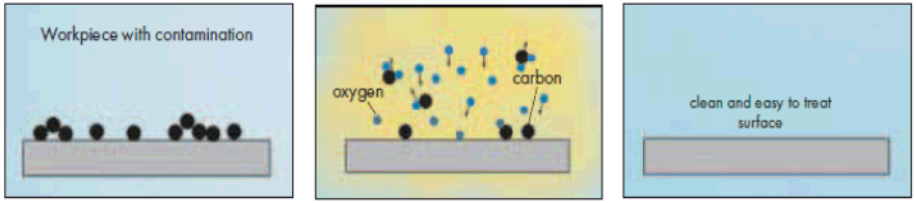
- Solution must be feasible
  - Can be used at needed point in process
  - Little special equipment or tooling
  - Little special training required
  - Can be focused on area needed
  - Affordable
  - Low risk to items being cleaned
  - Low hazard to worker

Constraints

- Identify most probable primary contaminants
  - Residue from cleaning circuit boards (gloves and flux in alcohol)
  - Silicone mold release (proximity contamination)
  - Atmospheric contaminants, handling, transportation (hydrocarbons)
- Identify possible cleaning method
  - Use at point of need on production floor
  - Applicable to soil types without harm to part
  - Can be focused to small area

Solution: Atmospheric Pressure Plasma Jet, aka Plasma Pen

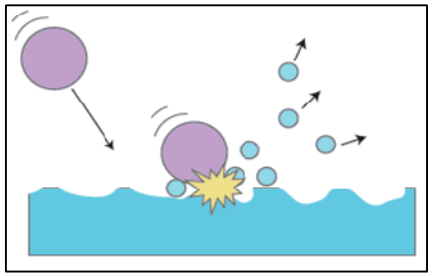
- Theory of Plasma: electrical energy ionizes gas
- Plasma cleaning due to etch



Oxygen plasma chemically interacts with organic contamination (hydrocarbons from handling and atmospheric exposure) forming species such as water, carbon monoxide and carbon dioxide. These species desorb from the surface, helped by the mild heating accompanying the plasma.

Solution: Atmospheric Pressure Plasma Jet, aka Plasma Pen (cont.)

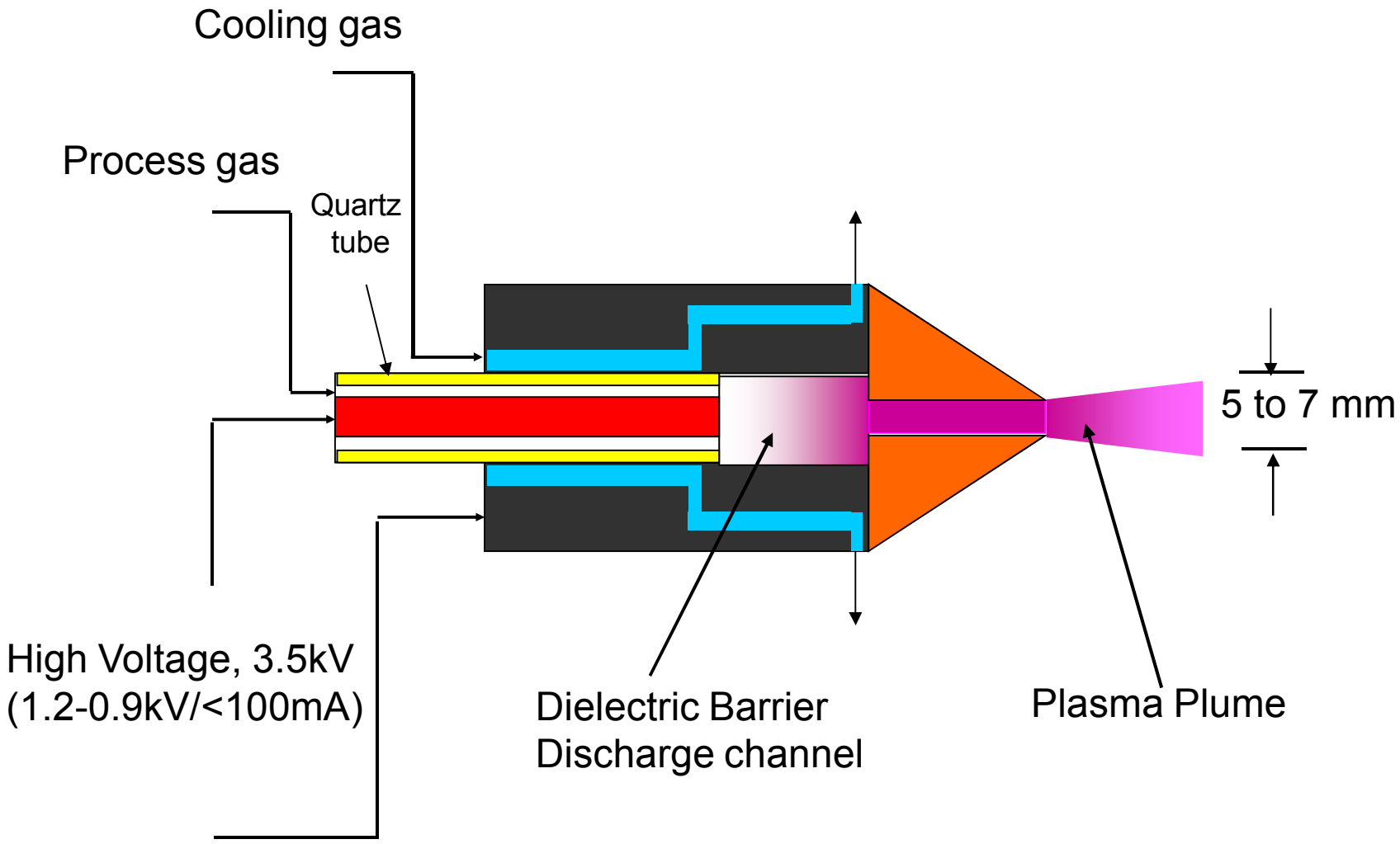
- Physical peening with energized ions to mildly sputter surface residue



Physical impact from energized ions breaks up and removes contaminants from surface. For atmospheric plasma jets the jet gas flow can help carry the molecules away to prevent recontamination.

Small spot size cleans local area without effecting surrounding material.

- Plasma Plume with small spot size and low heat
- <$20K, bench top



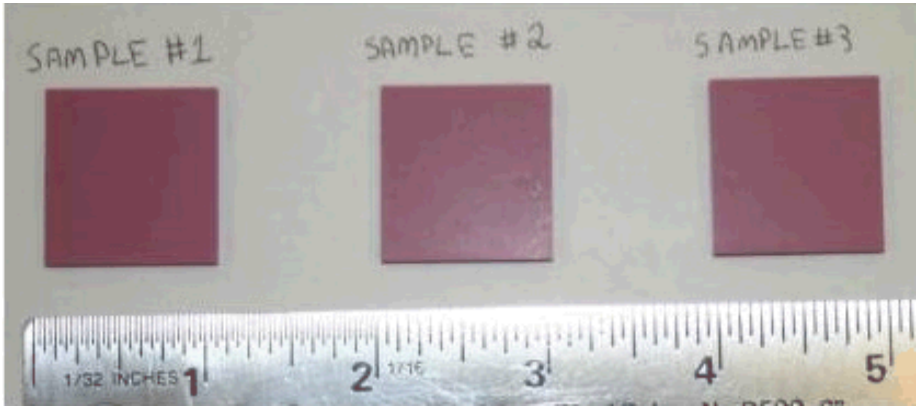
Plasma pen dielectric barrier discharge operation. High voltage AC discharges between two electrodes separated by an insulating dielectric.

Approach: Determine parameters for low risk local area atmospheric plasma clean

- Negligible Surface Modification
  - No reduction of glassy phase observed
  - No elemental content change
- Heat remains below current process parameters
  - Surface heating risk
  - Bulk heat transfer risk
- Effective cleaning
  - Determine size of effected area
  - Define application parameters
- Content and amount of cleaning residue

Test for desired parameters: Prepare samples

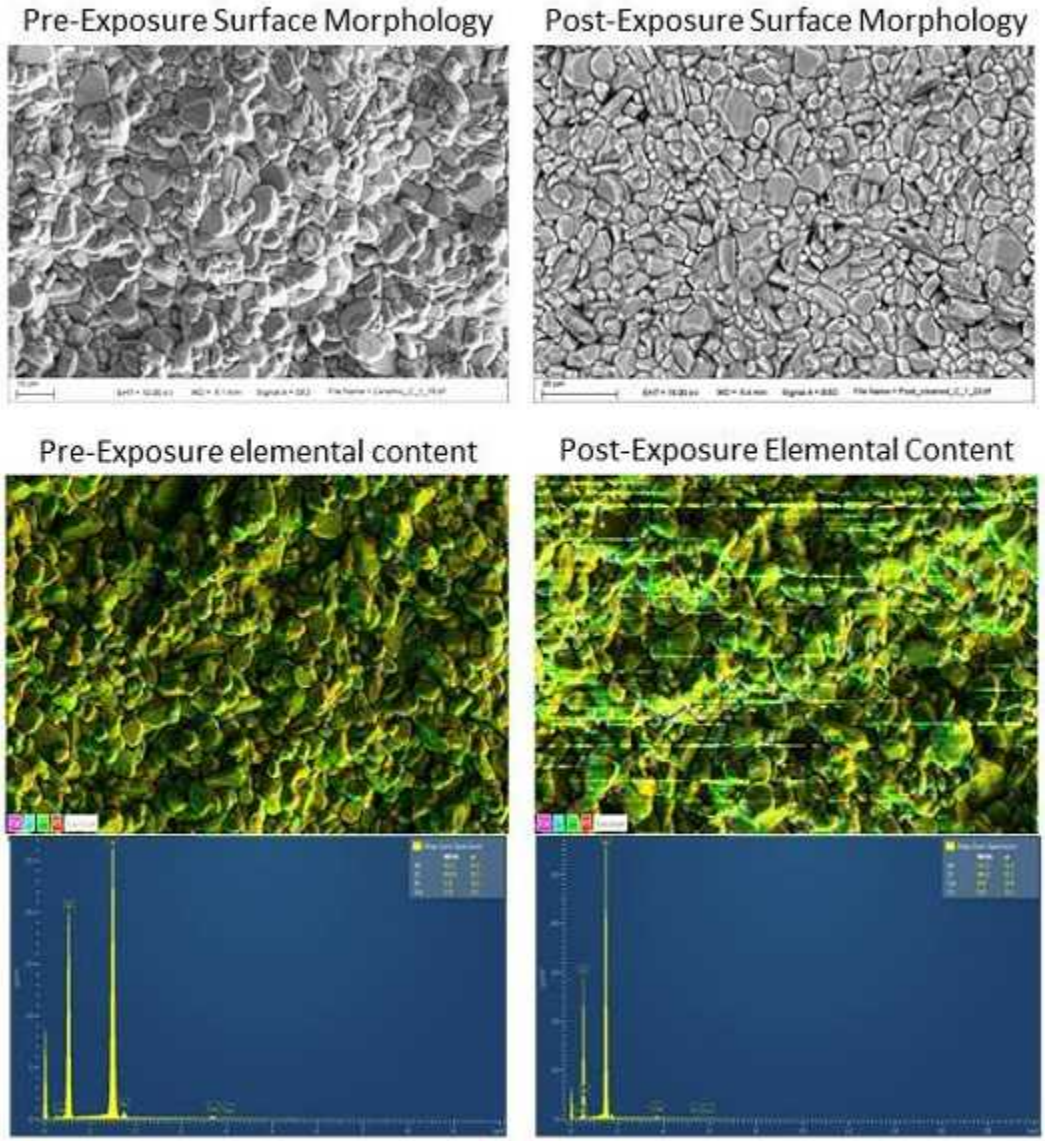
- Obtain same material in coupon form
  - Processed as closely to the actual part as possible
- Use production methods to prepare
  - Clean with same procedure and chemicals
  - Create contamination residue using production materials
    - Nitrile gloves worn while scrubbing soldered board with alcohol
    - Exposed to lab air for a week
    - In mold release coated tray, 2 heat cycles + soak



Test coupons. 1 “ x 1” squares and 1” diameter disks of 94% alumina ceramic

Negligible Physical Surface Modification

- Surface imaging with SEM, EDS
  - No surface morphology damage with 90 second exposure at ½" working distance
    - No apparent change in appearance of glassy phase
    - No evidence of etching or smoothing
  - Negligible elemental content change

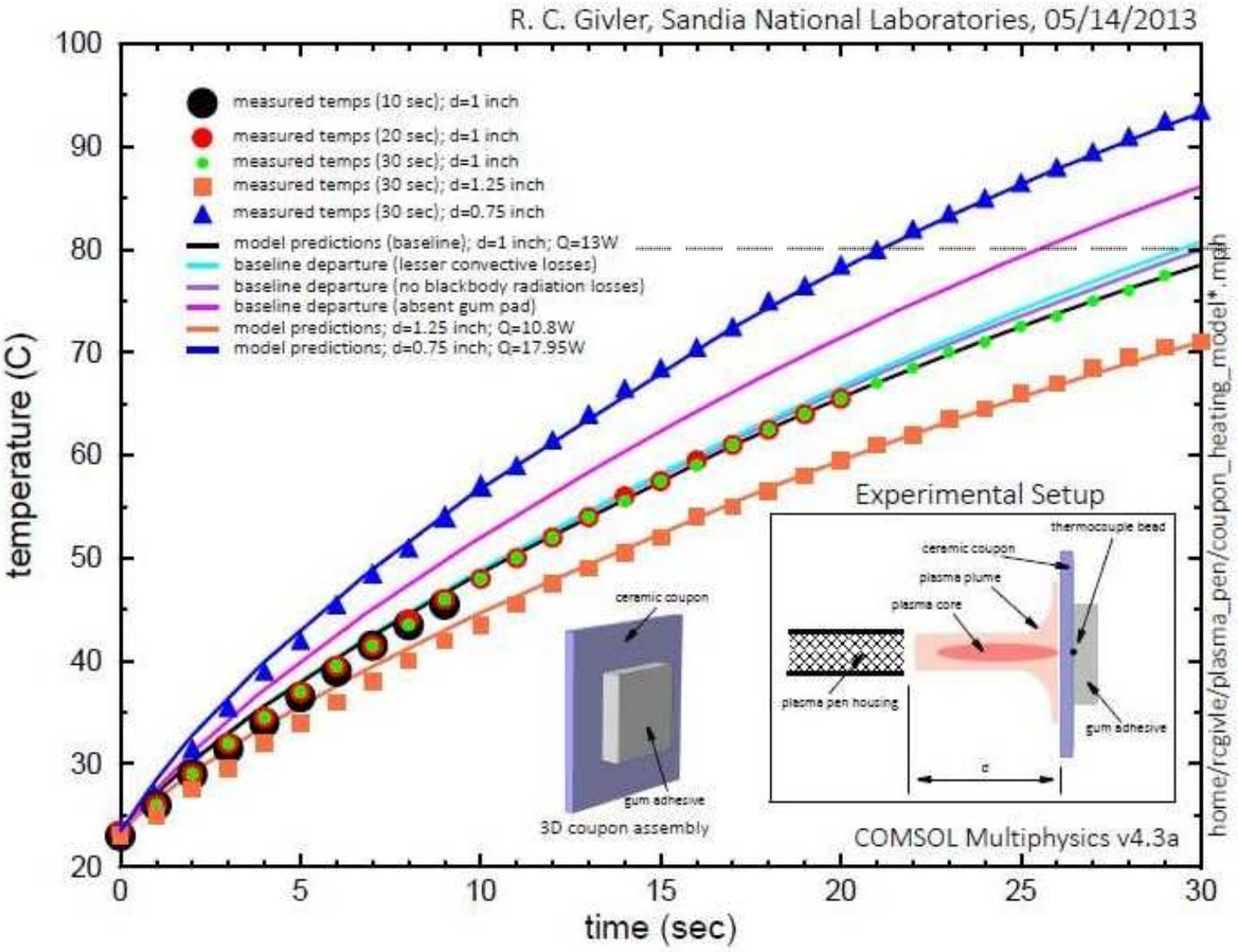


No change in grain size or structure is observed (SEM, Amy Allen, SNL)
Note: image on left is secondary electron, image on right is backscatter

No measureable difference in elemental content (center row and spectra in bottom row) is perceived (EDS, Amy Allen, SNL).

Negligible Physical Surface Mod. (cont.)

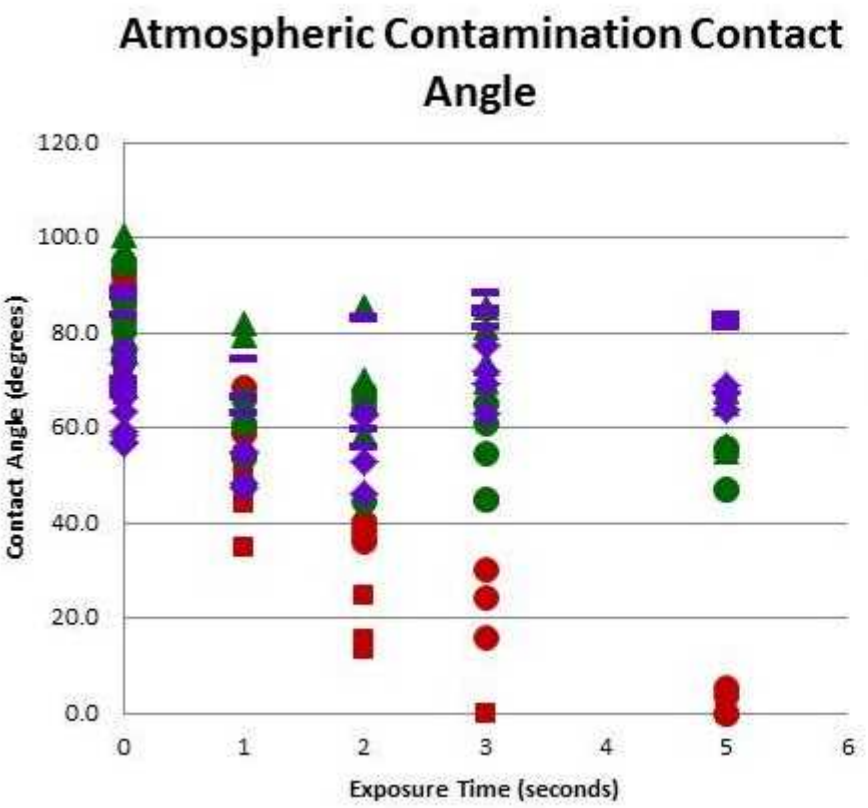
- Thermal measurement
  - Surface heating <80^{0}C on back of coupon (current process high temperature) desired
  - Heat transfer through 60 mil test coupon < 80^{0}C up to 20 seconds direct exposure at ¾”


R. C. Givler, Sandia National Laboratories, 05/14/2013
measured temps (10 sec); d=1 inch
measured temps (20 sec); d=1 inch
measured temps (30 sec); d=1 inch
measured temps (30 sec); d=1.25 inch
measured temps (30 sec); d=0.75 inch
model predictions (baseline); d=1 inch; Q=13W
baseline departure (lesser convective losses)
baseline departure (no blackbody radiation losses)
baseline departure (absent gum pad)
model predictions; d=1.25 inch; Q=10.8W
model predictions; d=0.75 inch; Q=17.95W
temperature (C)
time (sec)
100
90
80
70
60
50
40
30
20
0
5
10
15
20
25
30
Experimental Setup
thermocouple bead
ceramic coupon
plasma plume
plasma core
plasma pen housing
gum adhesive
d
ceramic coupon
gum adhesive
3D coupon assembly
COMSOL Multiphysics v4.3a
home/rcgivle/plasma_pen/coupon_heating_model*.mph

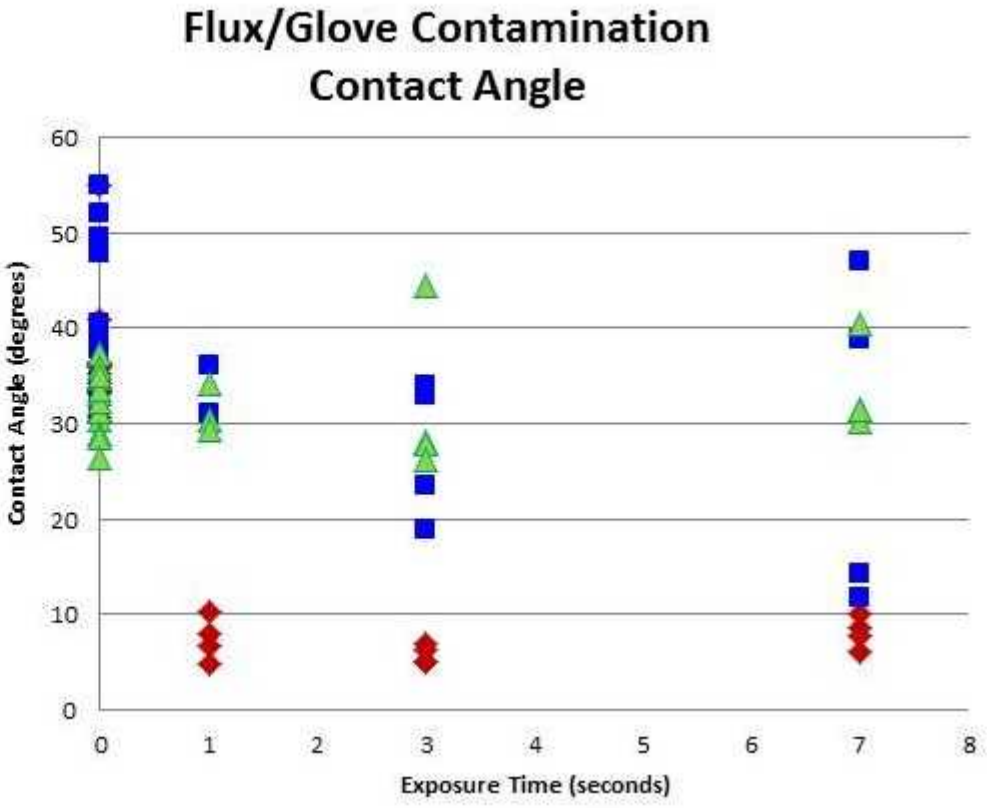
Cleaning Efficacy

- Wetting by water contact angle
  - Lower contact angle indicates superior bond quality
  - Determine optimal distance and exposure time using wetting angle
- Surface analysis to determine cleaning residue
  - Time of Fight Secondary Ion mass Spectroscopy (ToF-SIMS)
  - X-Ray Photoelectron Spectroscopy (XPS)

Contact Angle

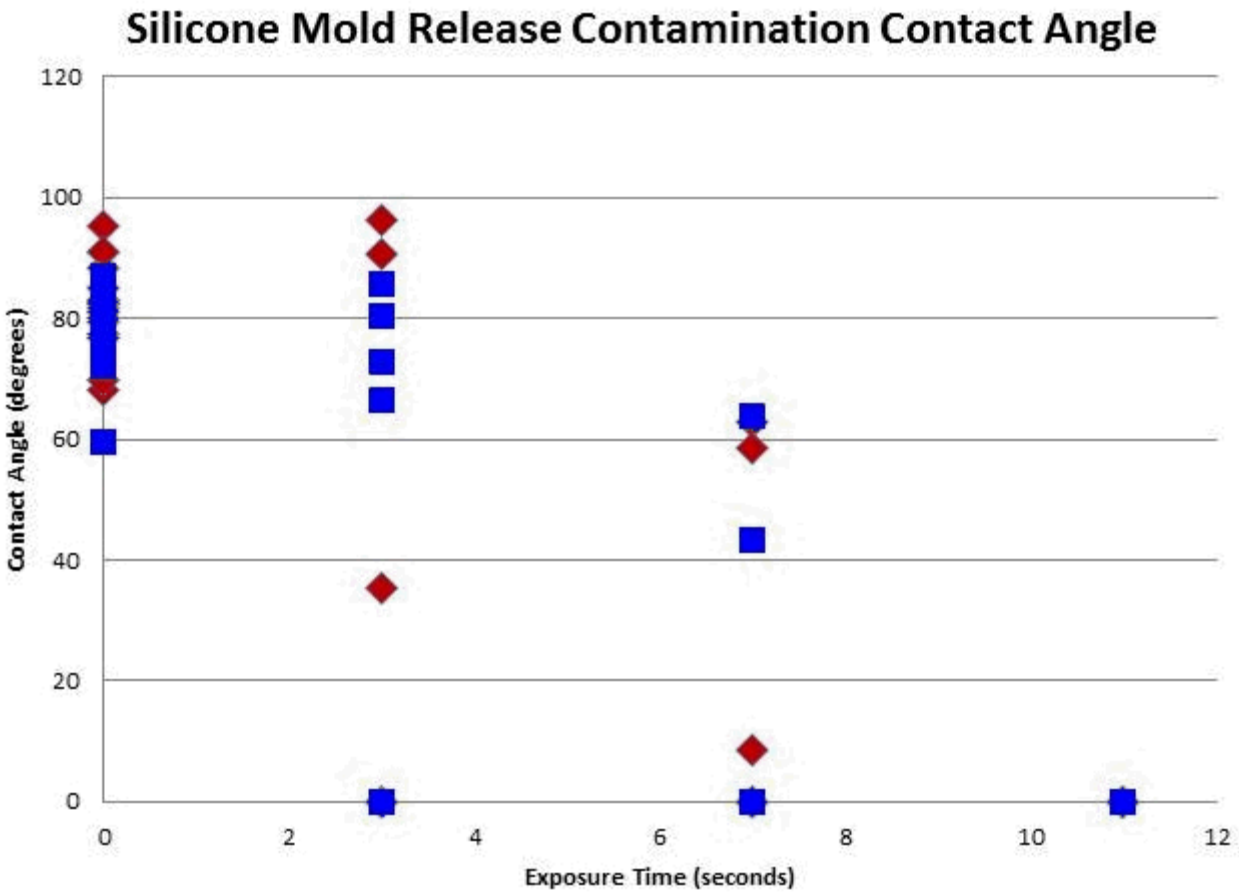


Distance of exposure:
red=½”, green=1”, purple=1½”



Distance of exposure :
red=1/2”, blue=3/4”, green=1”

Contact Angle

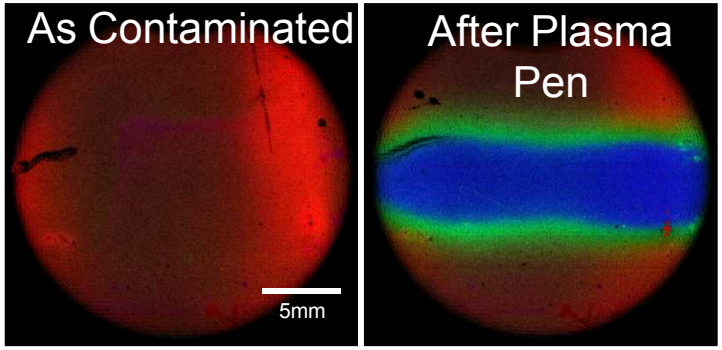


Distance of exposure: red=1/2”, blue=3/4”

DIFFICULTY: contamination coatings are non-uniform, adding variability to wetting

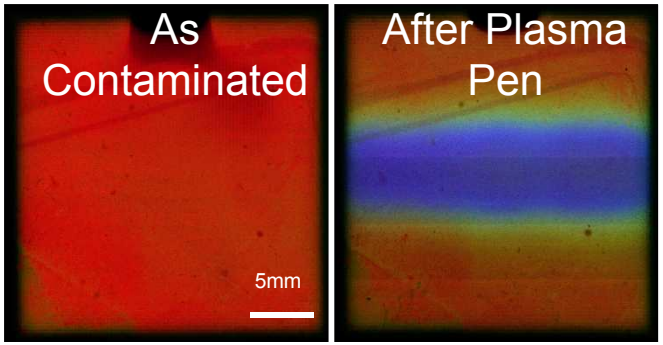
Cleaning Efficacy (cont.)

- Surface analysis to determine residue after plasma exposure, and spot size of sufficiently exposed area
  - ToF-SIMS



Red: Silicones
Green: Amines
Blue: Amines + Hydrocarbons

Plasma pen removes silicones (red), but also creates new surface chemistry (blue and green)

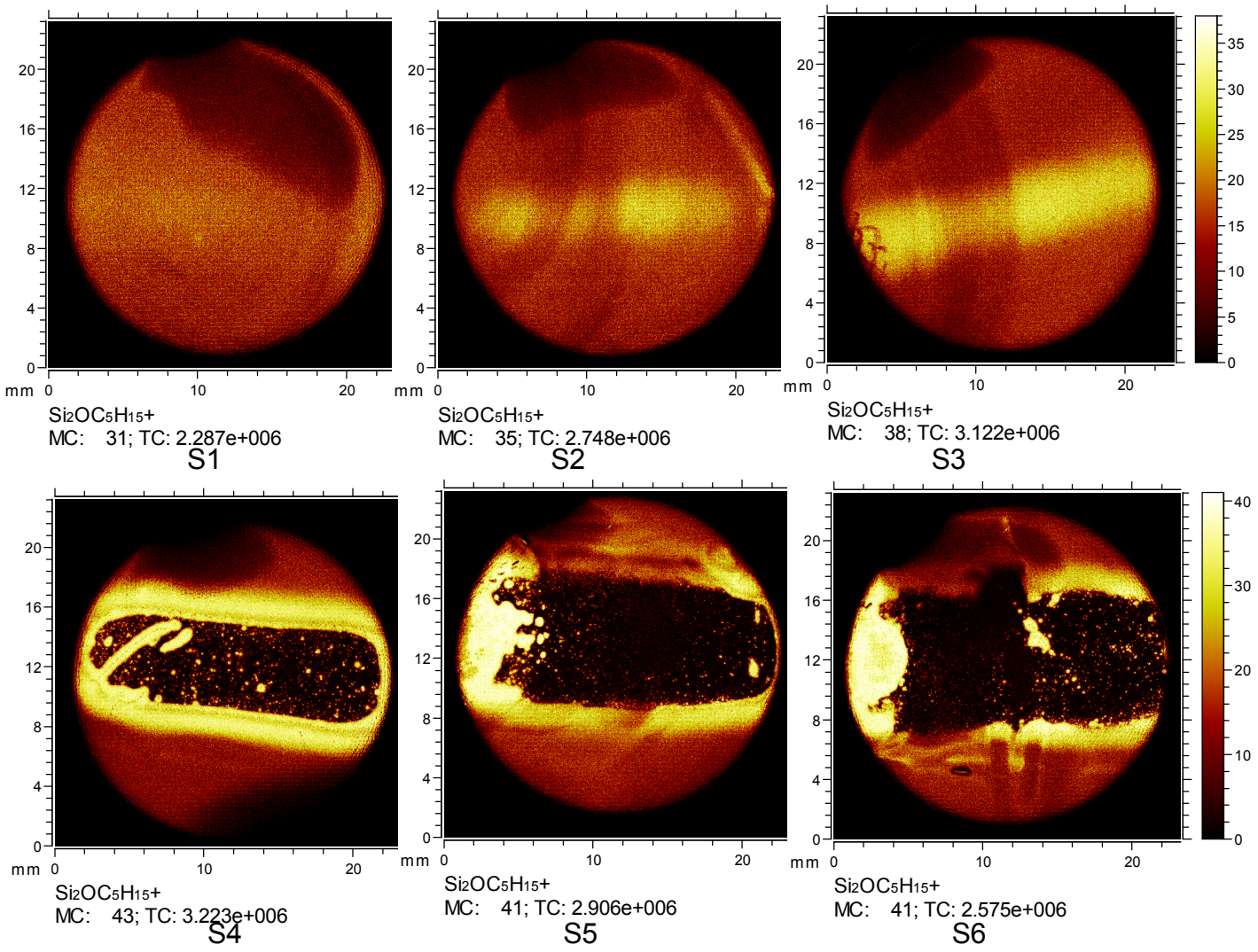


Red: Flux-Glove Residue
Green: Contaminants
Blue: Unknown new

Plasma pen removes residue (red), but also creates new surface chemistry (blue and green)

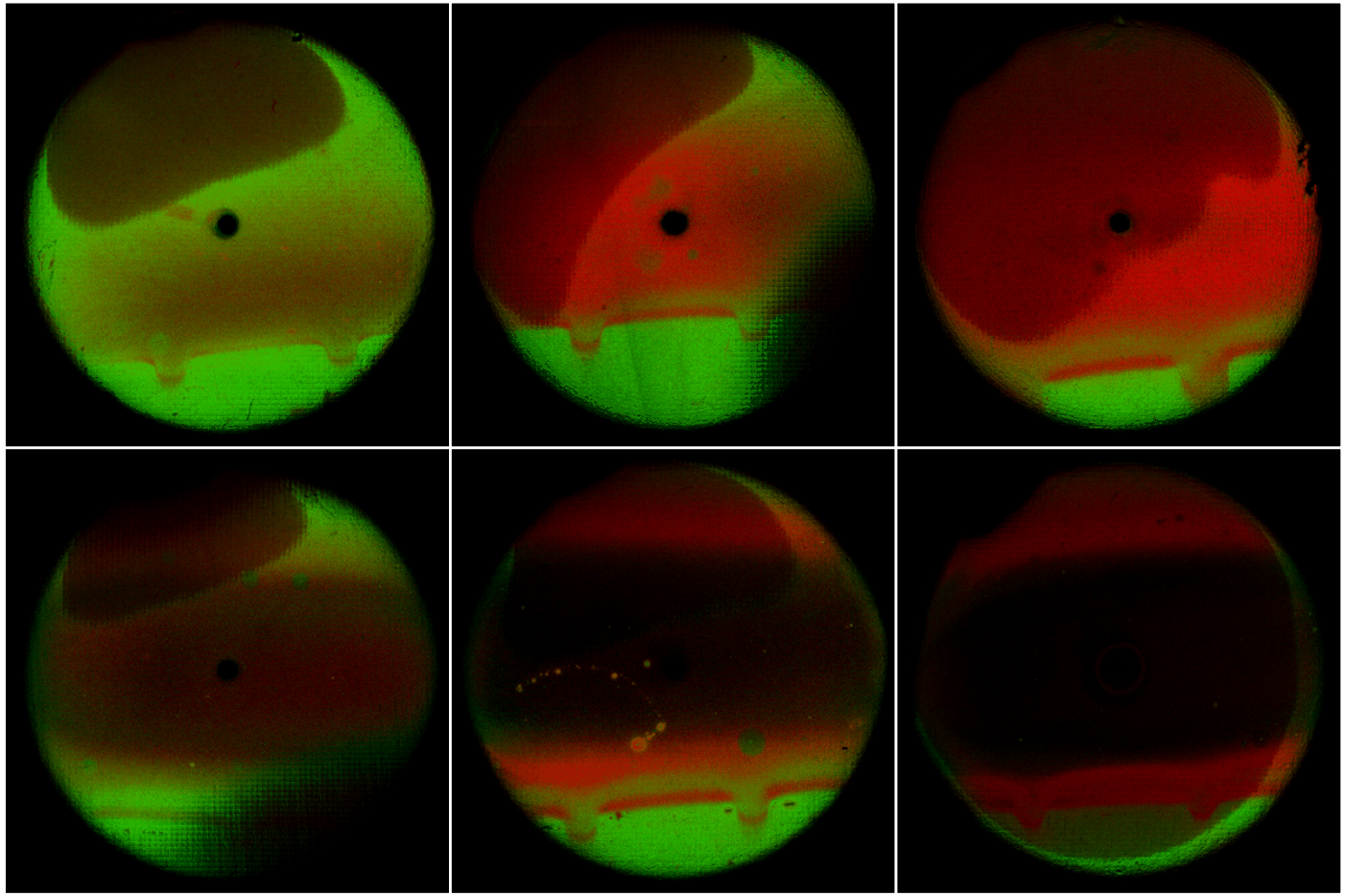
ToF-SIMS: Mold Release

Six 1” diameter ceramic disk coupons were exposed to silicone mold release. Each was then treated with the plasma pen, 3 at ¾” then 3 at ½” exposure distance with plasma dwell times of 10, 20 and 30 seconds per exposure distance.



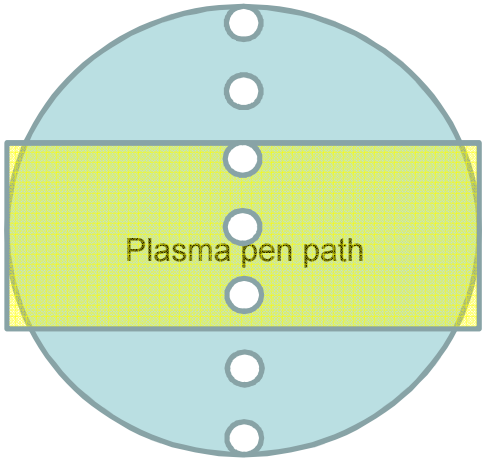
ToF-SIMS: Flux and Glove Residue

Six 1” diameter ceramic disk coupons were exposed to flux and glove residue in ethanol. Each was then treated with the plasma pen, 3 at ¾” then 3 at ½” exposure distance with plasma dwell times of 10, 20 and 30 seconds per exposure distance.



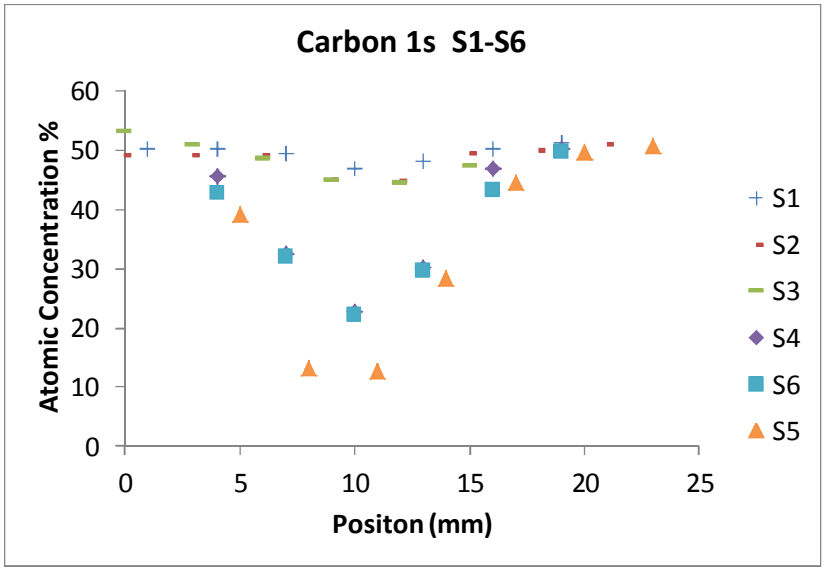
Cleaning Efficacy (cont.)

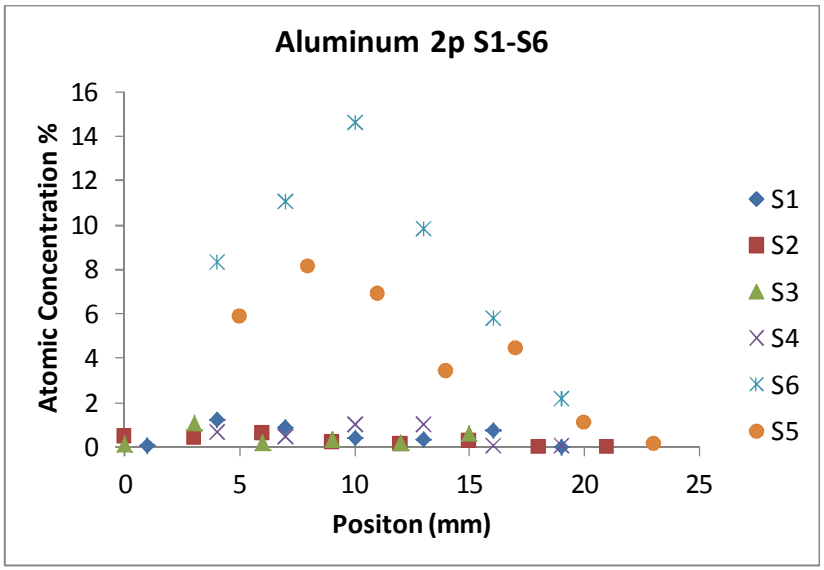
- Surface analysis to determine residue after plasma exposure, and spot size of sufficiently exposed area
  - XPS: locations analyzed every 3 mm perpendicular to plasma pen path

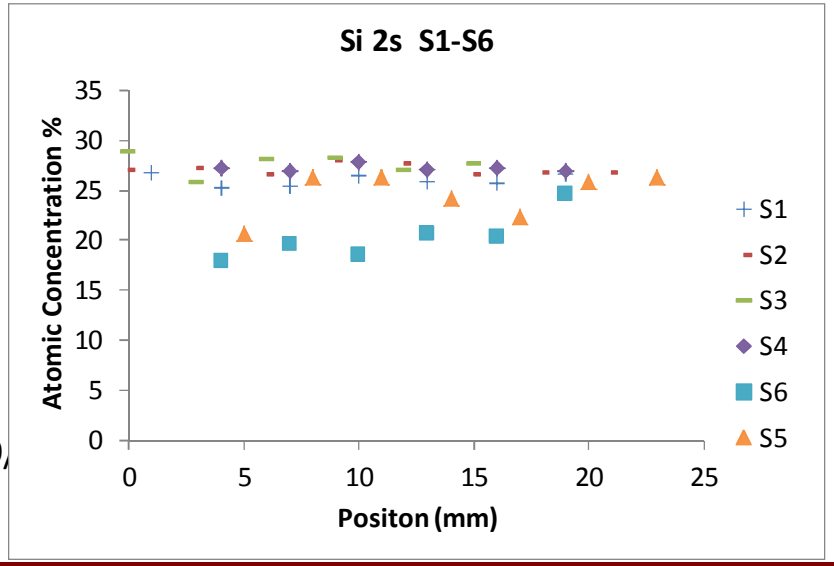


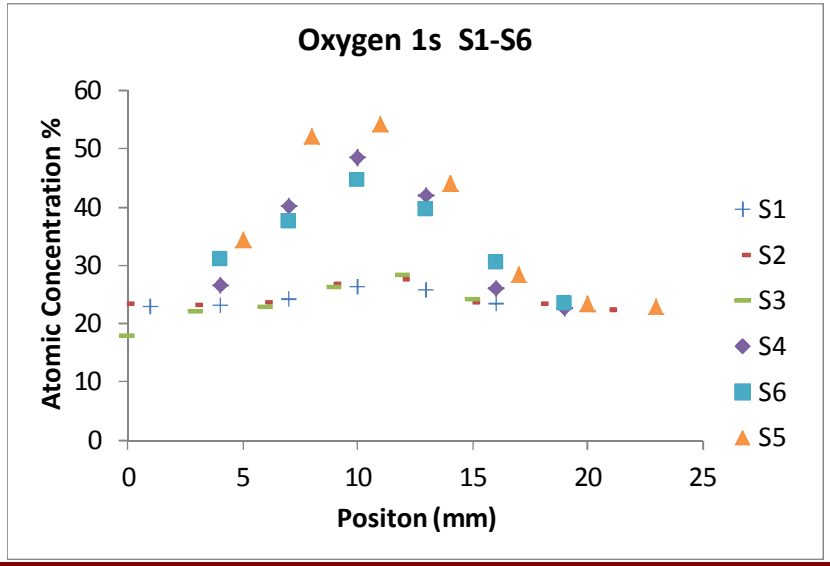
XPS data shows contamination layer thickness is little changed from treated surface to untreated surface

XPS: Mold Release

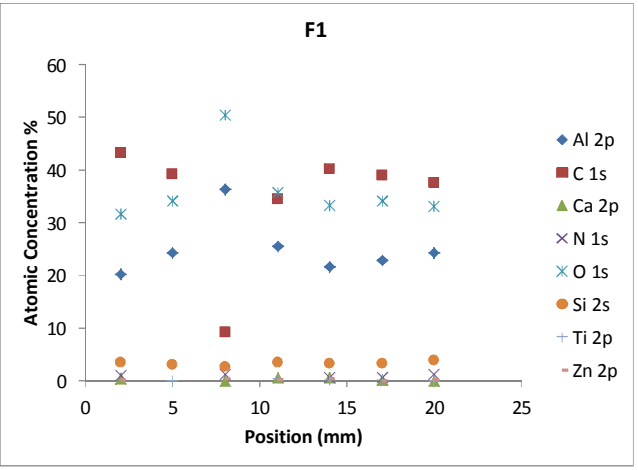


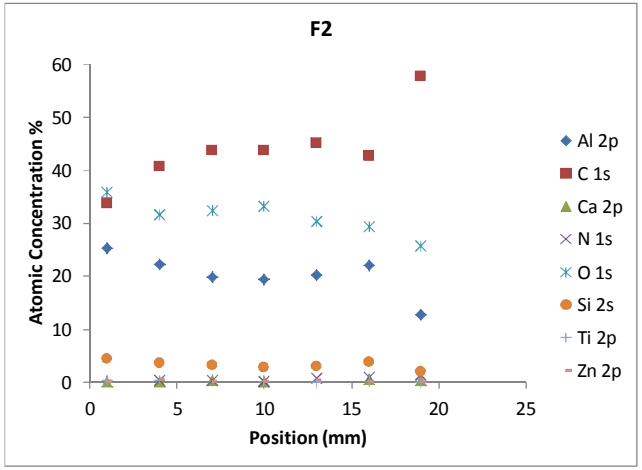


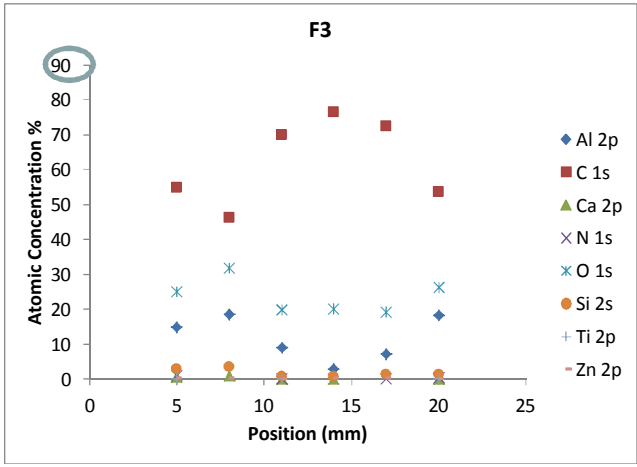


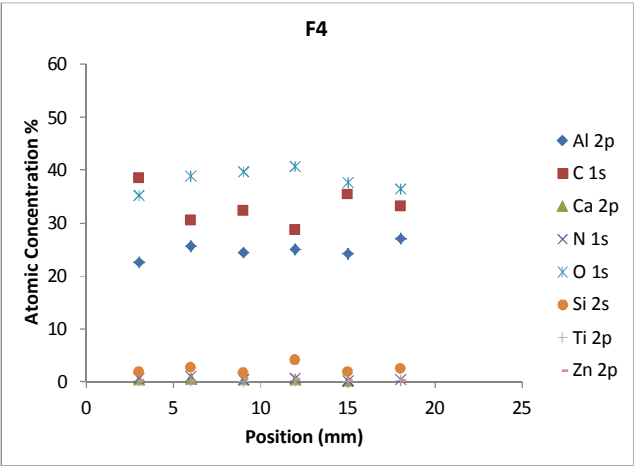


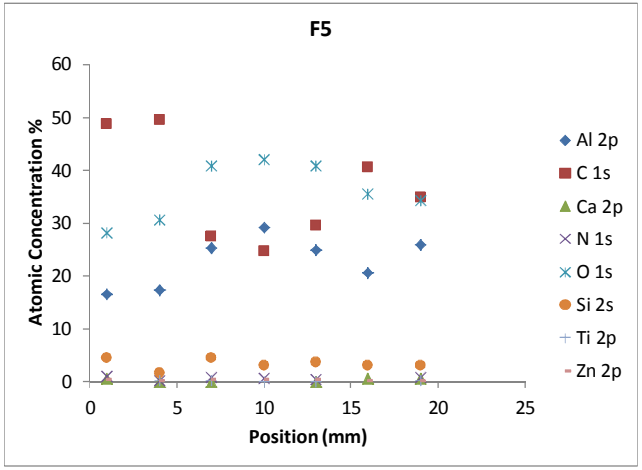
XPS: Flux and Glove Residue

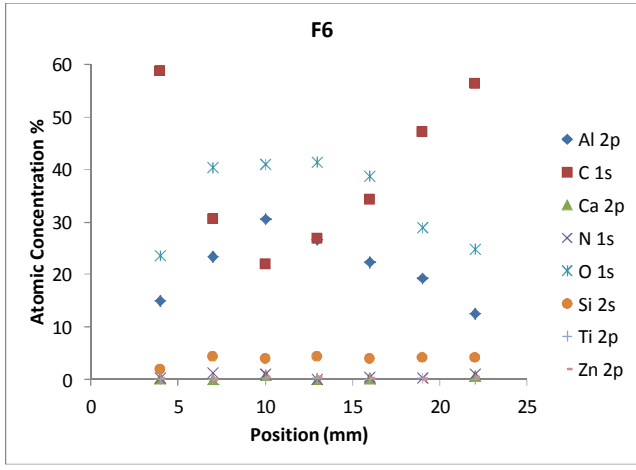












Results

- Low risk of damage to part from plasma and $\geq$ ½" working distance
- Effective cleaning distance is ½" separation from pen to surface
- Estimated 15 s/in^2 exposure time for cleaning below heat threshold
- Inconclusive cleaning with respect to residue

Future Work (Plasma Pen Characterization for Ceramic Substrate, Part 2)

- Surface analysis to identify chemistry changes and evaluate time required for sufficient cleaning
- Thermal modeling for part geometry and heat transport to most vulnerable surfaces
- Correlate previous test results to bonding strength of encapsulant on ceramic material

Acknowledgements

- Appreciation to:
  - Buddy Anderson
  - Michael Chance
  - David Barringer
  - Chip Olson
  - Richard Quintana

Thank You