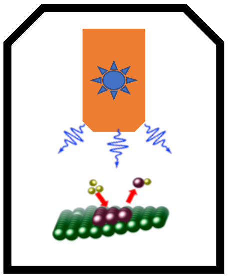
- Intellectual property decision group

Invention Report : 20210814

Hydrocarbon Mitigation System (HMS)
for Electron Microscopy

NESTOR J. ZALUZEC
Sr. Scientist - PSC

20220928
Argonne National Lab.



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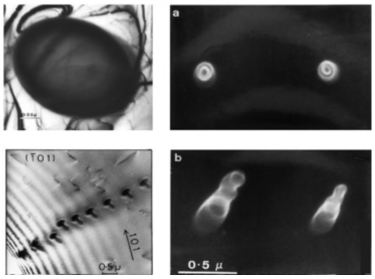
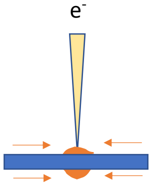
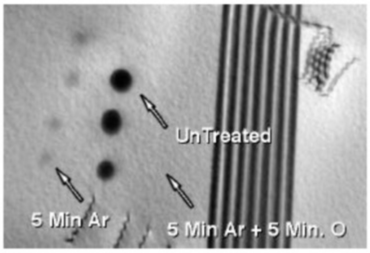
PRIOR ART: Background - Hydrocarbon Mitigationfor Electron Microscopy

In 1995, ANL developed the plasma cleaner (U.S. Pat. # 5,510,624) for reduction/removal of hydrocarbon contamination in robust materials and instruments by using reactive gas plasma's for use in electron microscopy. The contamination is formed by surface hydrocarbons migrating to the electron probe.







Examples of " mobile hydrocarbon contamination"

Before / After Plasma cleaning

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Licensee's of the ANL Plasma Cleaning Technology for Electron Optical Beam Lines

South Bay Technology
Fischione Instruments
XEI Scientific
Structure Probe Inc
Gatan Inc.
Zyvex Group.

- Invented the Technology
- Proved the Methodology
- Advised Manufacturers on design
- Tested products as part of the license agreements

South Bay Technology · SPI Supplies · Fischione Instruments · XEI Scientific, Inc. · gatan · zyvex technologies

Six companies selling in the USA licensed this. Non-US companies did notlicense as the no international filing were done.

Units sell for $50-125K. Number of units sold ? Every Materials Science EM facility I have visited has at least one.

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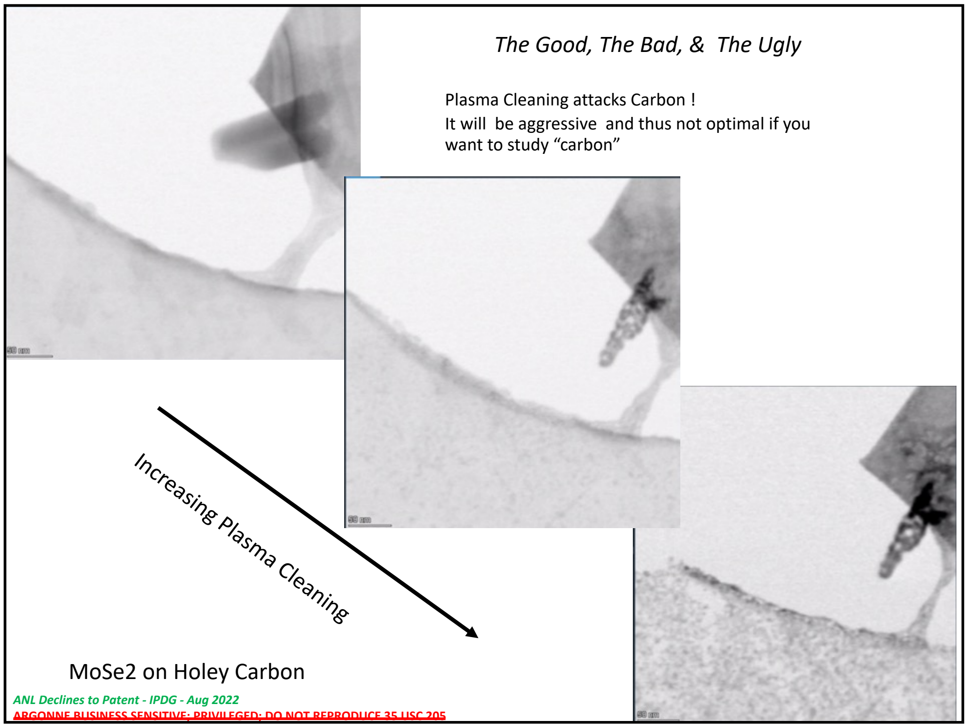
The Good, The Bad, & The Ugly

Plasma Cleaning attacks Carbon !
It will be aggressive and thus not optimal if you want to study "carbon"



MoSe2 on Holey Carbon

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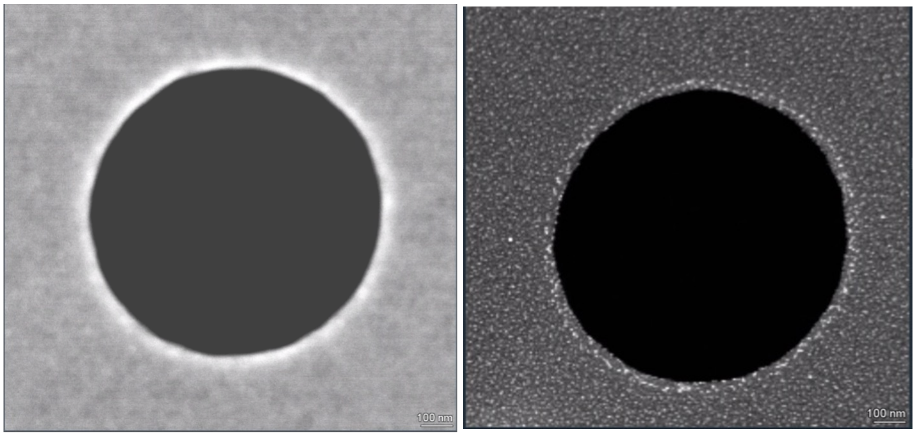
The Good, The Bad, & The Ugly

Plasma Cleaning attacks Carbon !



Increasing Plasma Cleaning

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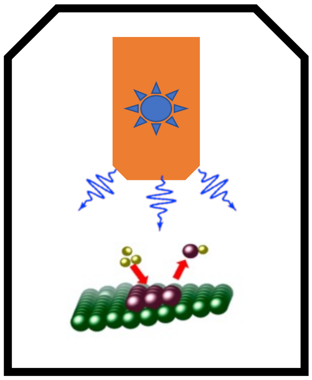
New Invention Report : 20210814

Hydrocarbon Mitigation System (HMS) for Electron Microscopy



A new method has been developed which can be used which is gentler, and significantly less expensive (< $1000) than plasma cleaning. It is applicable to specimens which are *lightly contaminated* with mobile surface hydrocarbons such as Graphene and carbon nanotubes, organic films which are be significantly affected by an energetic plasma.

Similar principle to Plasma cleaning but without the plasma!

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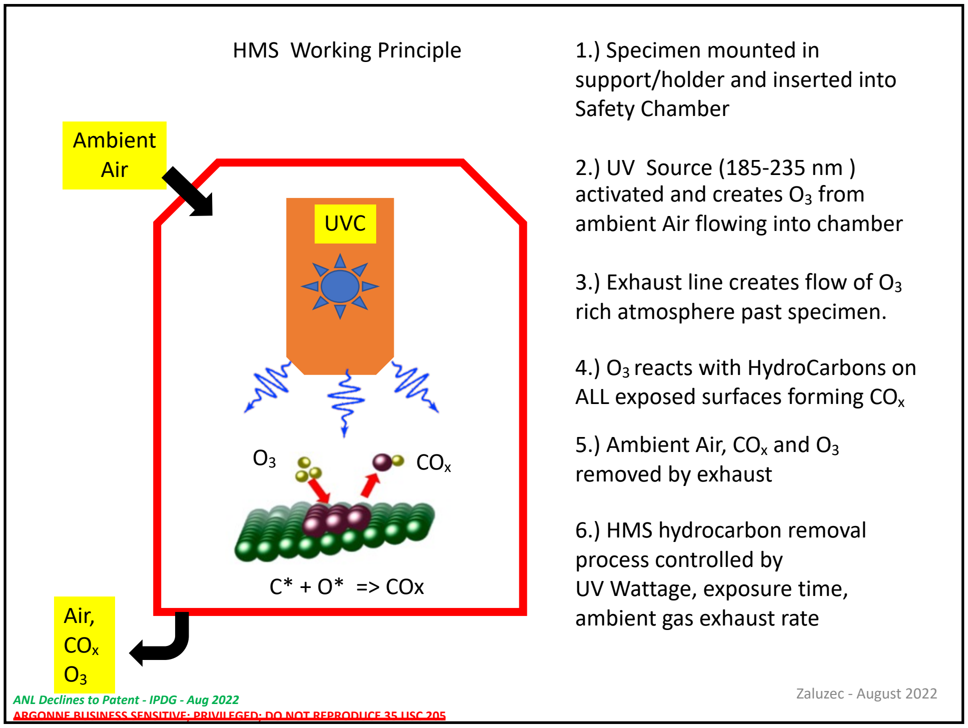

HMS Working Principle

1.) Specimen mounted in support/holder and inserted into Safety Chamber

2.) UV Source (185-235 nm) activated and creates O_3 from ambient Air flowing into chamber

3.) Exhaust line creates flow of O_3 rich atmosphere past specimen.

4.) O_3 reacts with HydroCarbons on ALL exposed surfaces forming CO_x

5.) Ambient Air, CO_x and O_3 removed by exhaust

6.) HMS hydrocarbon removal process controlled by
UV Wattage, exposure time,
ambient gas exhaust rate

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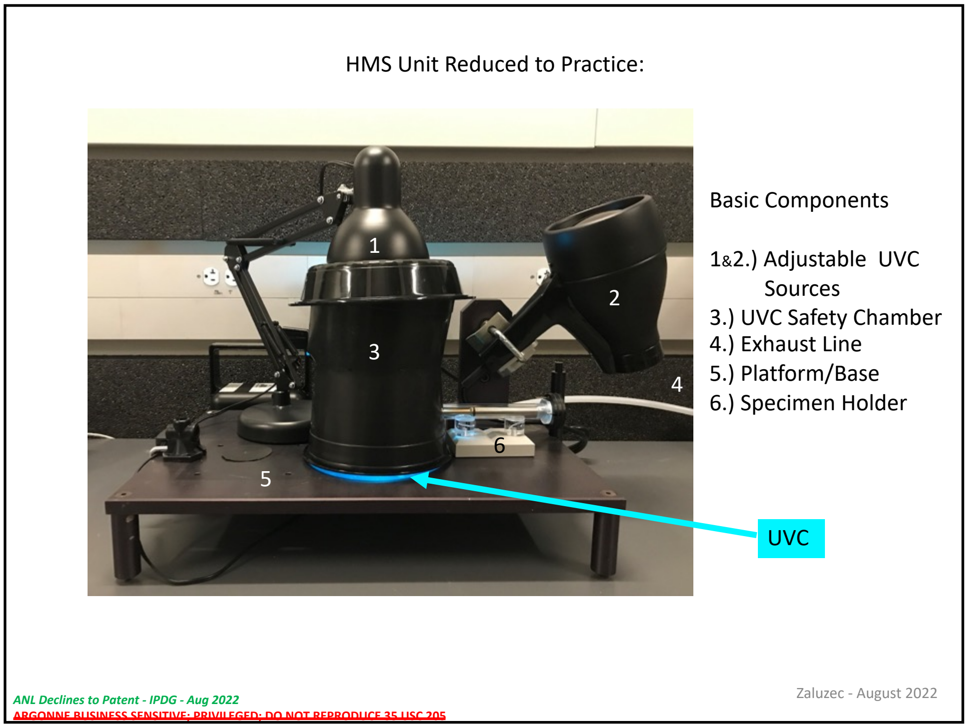

HMS Unit Reduced to Practice:

Basic Components

1&2.) Adjustable UVC Sources
3.) UVC Safety Chamber
4.) Exhaust Line
5.) Platform/Base
6.) Specimen Holder

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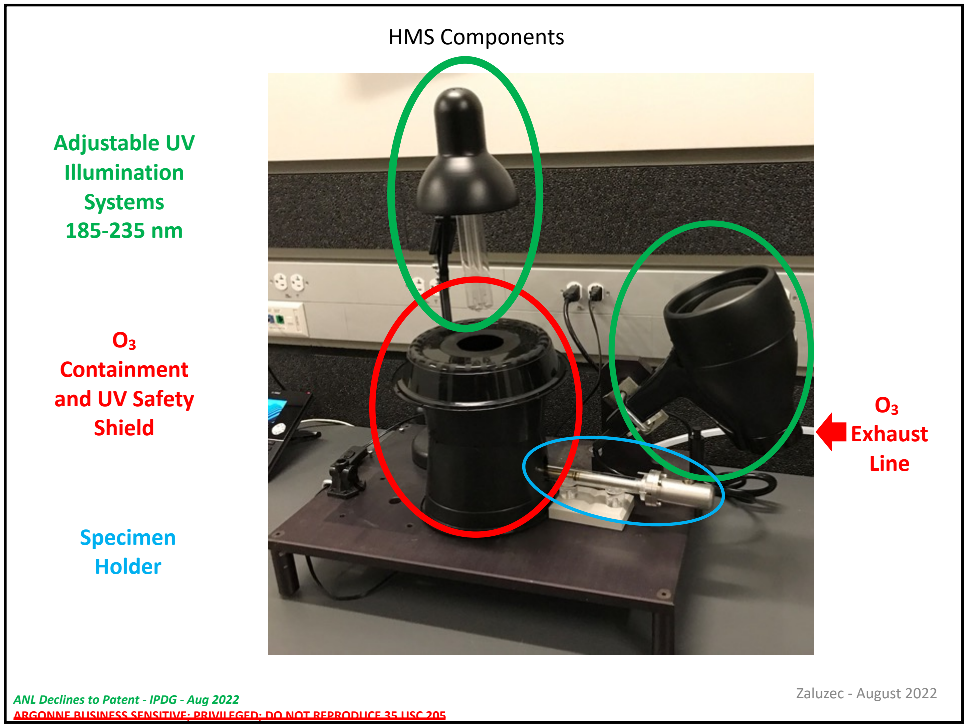

HMS Components

Adjustable UV Illumination Systems 185-235 nm

O_3 Containment and UV Safety Shield

Specimen Holder

O_3 Exhaust Line

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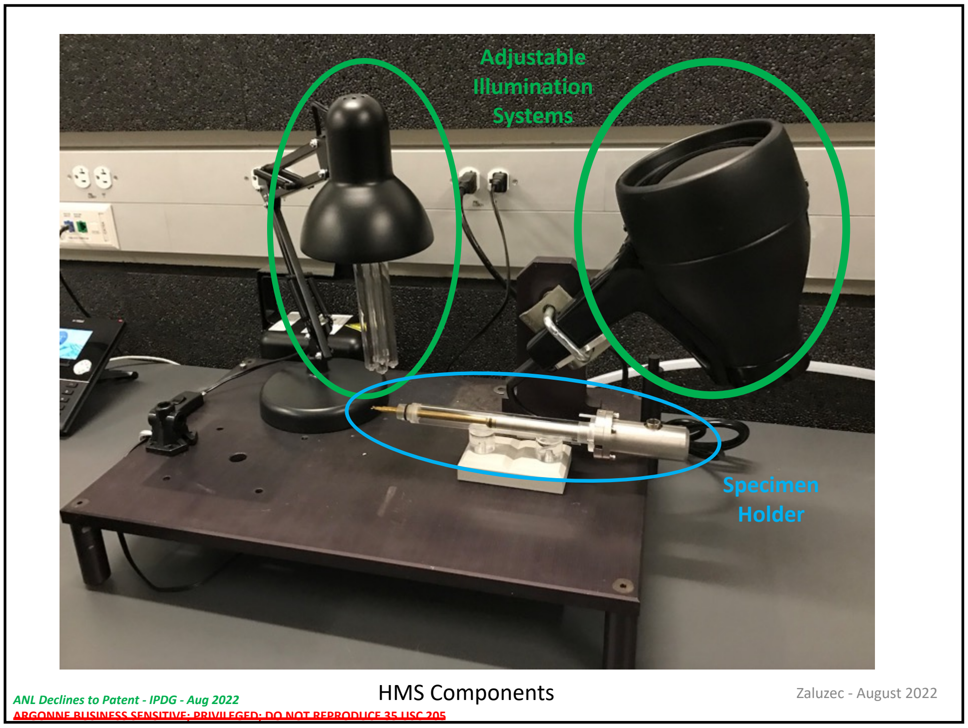

Adjustable Illumination Systems

Specimen Holder

HMS Components

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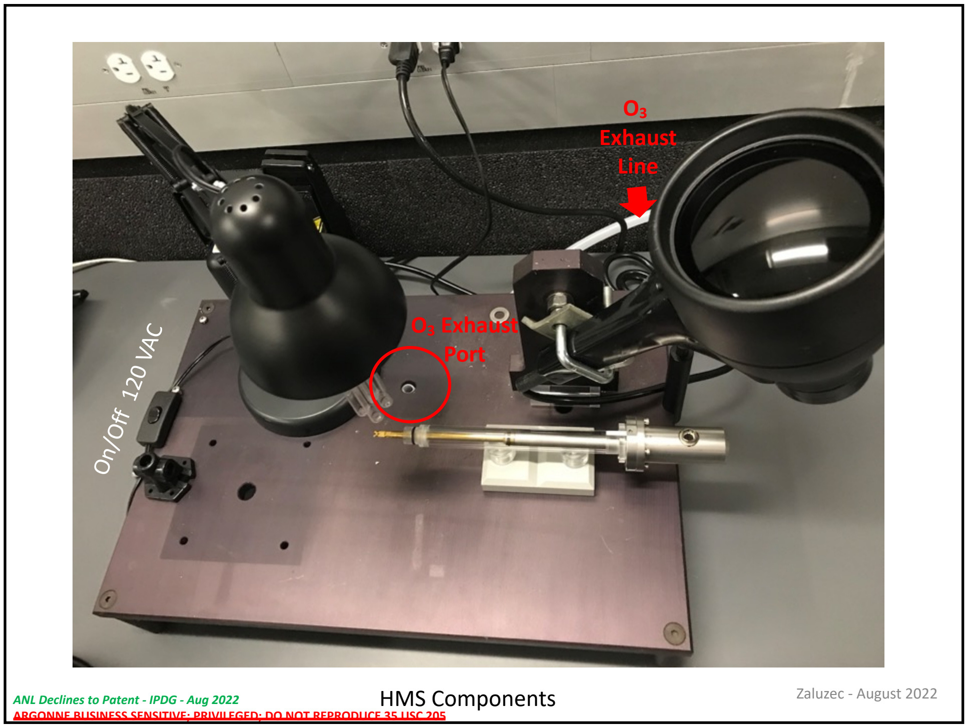

HMS Components

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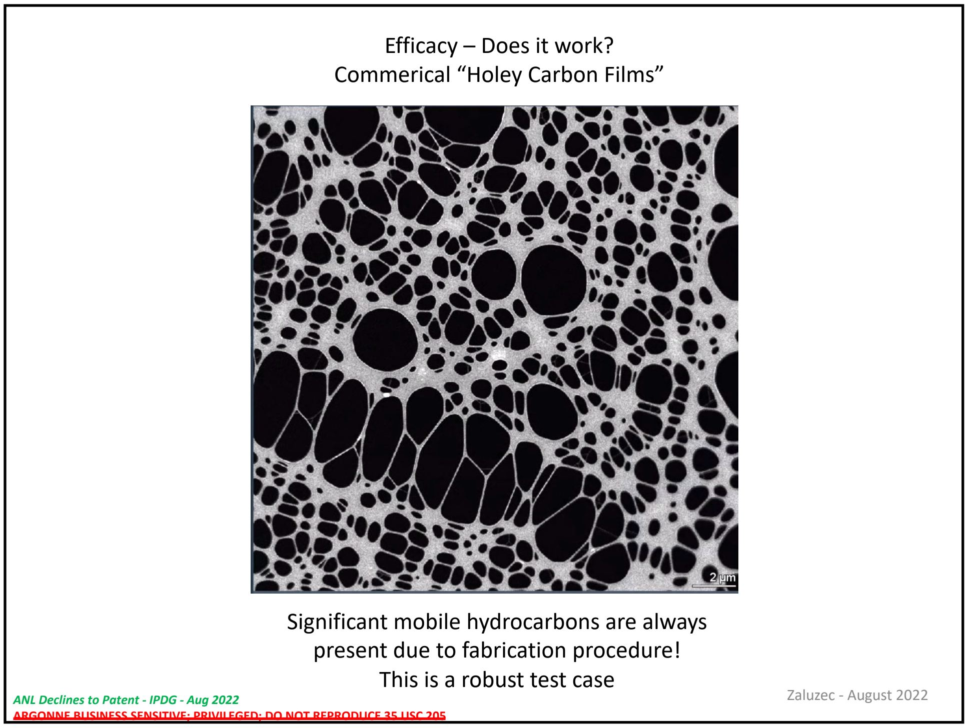

Efficacy – Does it work?
Commerical “Holey Carbon Films”

Significant mobile hydrocarbons are always
present due to fabrication procedure!
This is a robust test case

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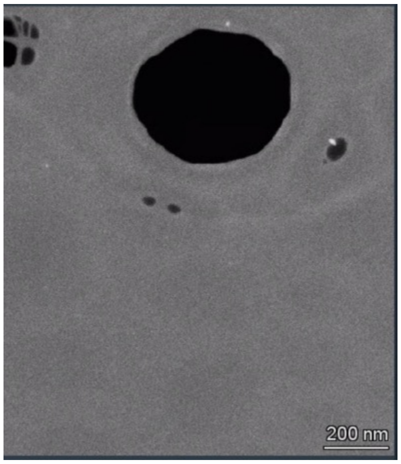
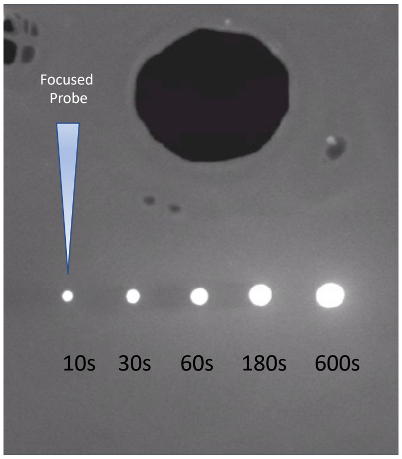
Does this work? Pre UVC Treatment

Before



”Fresh (with mobile surface HC) Specimen ”

After Focused Probe



Post Spot Mode HC Accumulation

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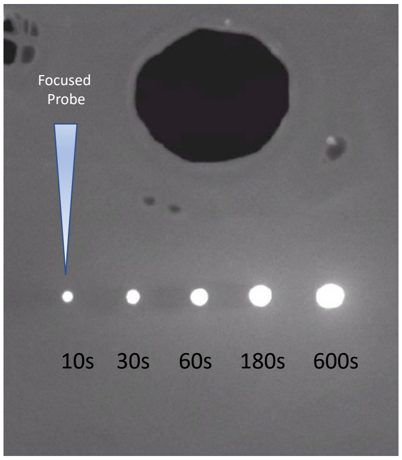
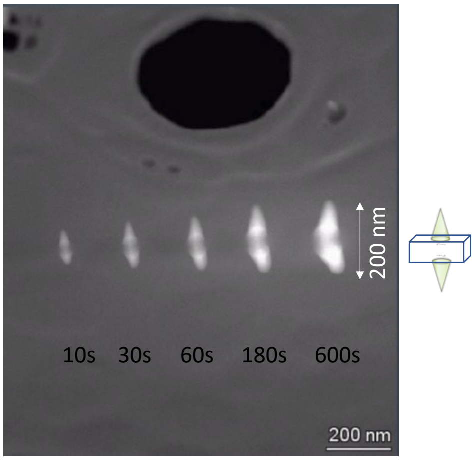
Does this work? Pre UVC Treatment

After Focused Probe



Pre UVC



Pre UVC tilted 30 degrees

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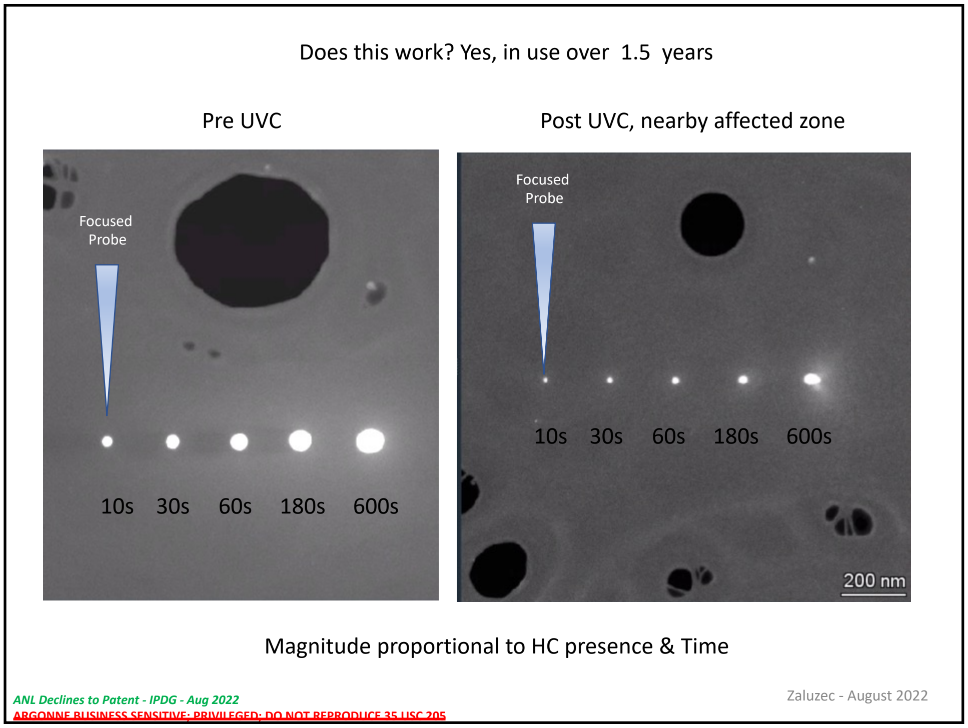

Does this work? Yes, in use over 1.5 years

Pre UVC

Post UVC, nearby affected zone

Magnitude proportional to HC presence & Time

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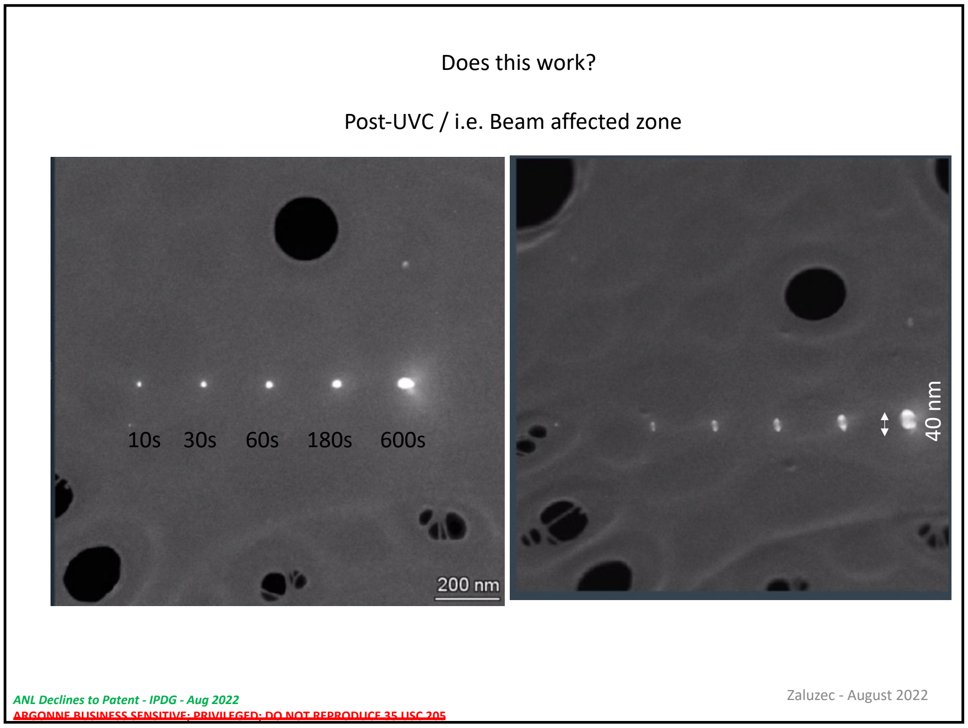

Does this work?

Post-UVC / i.e. Beam affected zone

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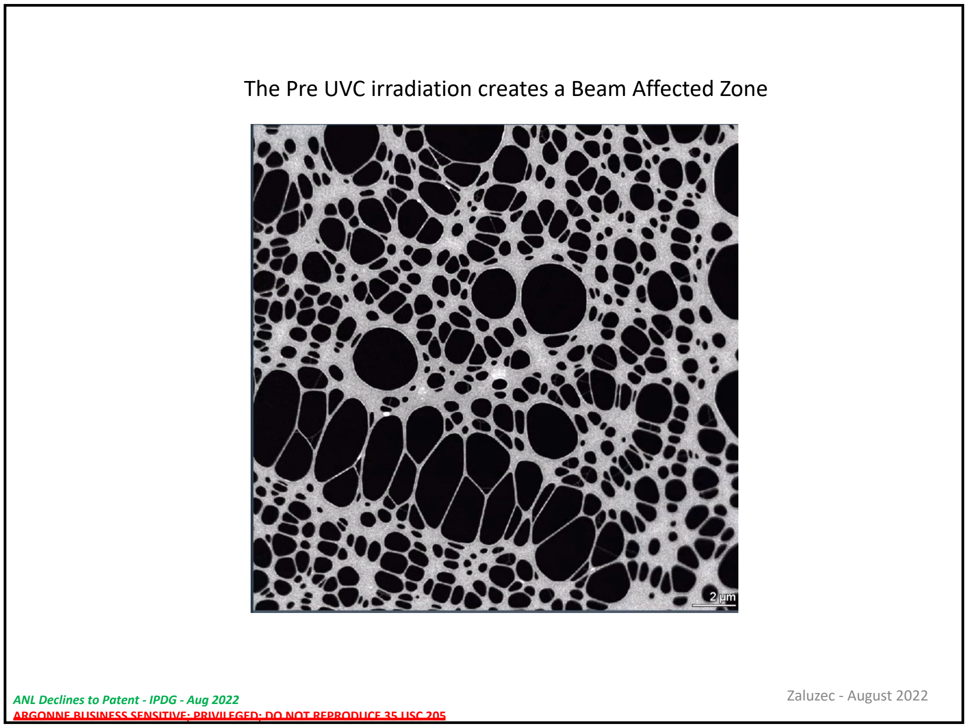

The Pre UVC irradiation creates a Beam Affected Zone

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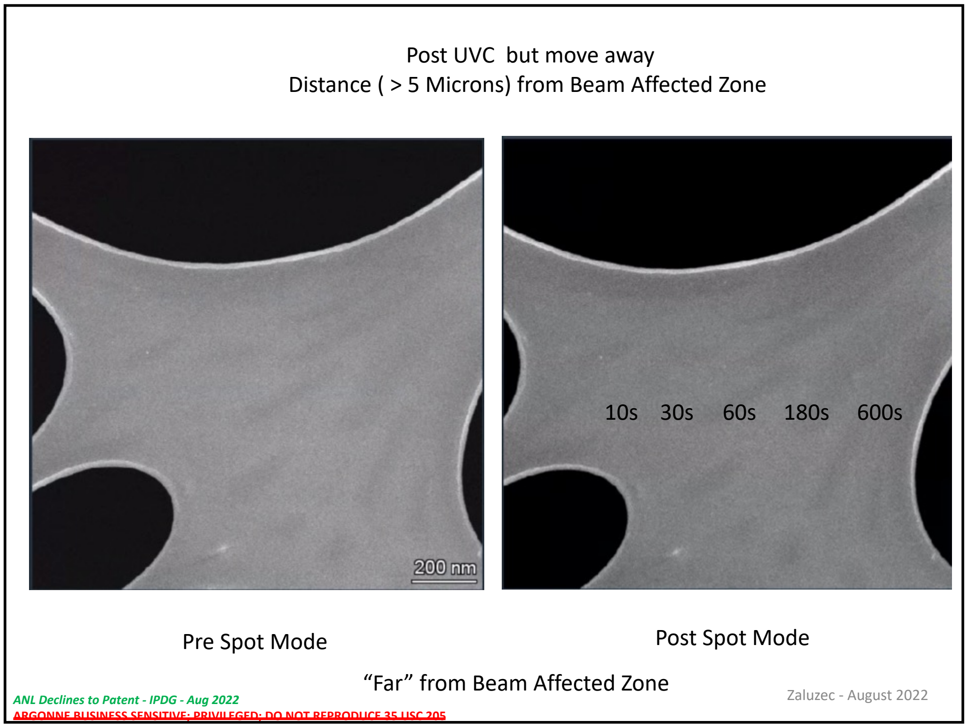

Post UVC but move away
Distance (> 5 Microns) from Beam Affected Zone

Pre Spot Mode

Post Spot Mode

“Far” from Beam Affected Zone

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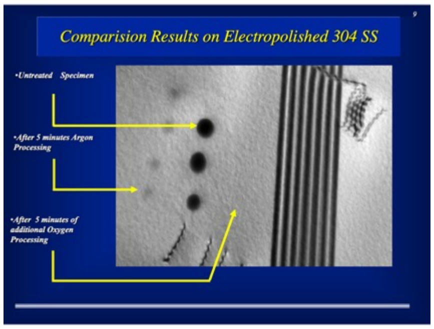
Which is Better:

Plasma Cleaning..... Robust metals, ceramics, very Thick materials

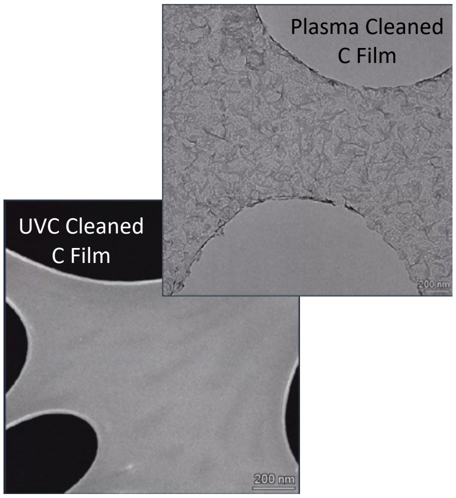
UVC – Light contamination, Carbonaceus Films, 2 D Materials (Graphene !), Soft Matter



Comparision Results on Electropolished 304 SS

- *Untreated Specimen*
- *After 5 minutes Argon Processing*
- *After 5 minutes of additional Oxygen Processing*

Plasma Cleaned Robust Metal



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Advantages
- Inexpensive, easy to implement.
- Works on “C” sensitive materials & not aggressive.
- Important for any “material” which may have HC’s involved
  - Energy Materials, Soft Matter , BioSystems

Commerical Interest
- Likely, have not talked about this to commercial vendors

  Plasma Cleaner Process is a commercial success but
  an IP Failure for the Lab due to insufficient protection

  Is this worthwhile..... $$$?

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