



## Linear Collider Collaboration Tech Notes

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### Plasma Discharge Cleaning of NLCTA Cells

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## Plasma-Discharge Cleaning of NLCTA Cells†

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### ‡Abstract

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### 1 Introduction

In-situ plasma discharge-cleaning is an accepted procedure for cleaning carbon from the walls of accelerator and storage ring sections, e.g. an Ar/5%O mixture was used to clean the B-factory copper High-Energy Ring (HER) beam chambers. In the case of copper, at least, the use of rather massive argon leads to surface roughening, not a particular problem for already-rough as-extruded surfaces. However, for the cleaning of mirror-smooth NLCTA cells, the roughening effect can be mitigated by the use of a lighter gas, e.g. pure hydrogen, in the discharge. The particular mechanisms for the discharge-cleaning process differ slightly in the two cases. With oxygen, the surface carbon is oxidized to CO and CO<sub>2</sub>, which is weakly-bonded to the surface (compared to elemental carbon), and easily desorbs. However, the underlying copper surface can also become oxidized as well in this process. With hydrogen, the surface carbon is hydrogenated to hydrocarbon molecules which are also weakly-bonded and desorb. The low mass of hydrogen does not momentum impact-roughen the surface and, in addition, hydrogen removes the thin surface oxide via the creation of desorbable water-hydroxyl molecules.

DC hydrogen plasma can be used to clean brazed NLCTA cell structures by placing the section into a vacuum system, with a central metal anode at the axis of the assembly. A potential is applied between the anode and the cell stack, hydrogen is flowed through and a cold

cathode discharge is triggered by a cosmic ray. The extent of the plasma glow is governed by the pressure and voltage according to the Paaschen Law so that, by varying the conditions, the discharge could be forced to penetrate to varying axial radii inside the structure. Surface contamination will be removed by the physical/chemical process described above. Particles, which usually stick to surfaces via an interfacial oxide or water (van der Waals bonding) or static charge, can be dislodged.

This idea is attractive. Following the manufacture of a cell stack assembly, the stack could be given a final, non-destructive, plasma-cleaning to remove debris and contamination. No residue of the cleaning process material, such as would happen with liquid cleaning, is left trapped to later degas during accelerator operation.

### 2 Experimental Details

A test chamber of about 10 liters volume was set up to accept a short vertical unbrazed thirteen-cell NLCTA stack. The cells were stacked individually so that cells along the stack could be removed for later analyses. To facilitate later analysis, some cells were very thinly slit-sawn, on the diameter, prior to cleaning in the SLAC Plating Shop. They could then be re-assembled without a gap into essentially complete cells.

During initial setup of the system, a few cells had “pie” wedges removed to aid viewing of the discharge that takes place inside the stack. Optimal discharge conditions of pressure, discharge current and voltage could then be chosen, by observation through a vacuum window, to fill the stack interior with luminescent discharge. Those conditions were 300 volts, 200 mA discharge current, 250 mT pressure and one hour cleaning time.

Cells were checked for cleanliness following processing in the SLAC Plating Shop, with process C01A (no Oxyban), by X-ray Photoelectron Spectroscopy (XPS). Two experiments were executed:

- 1) Thermally oxidize two cells in air at 250°C for two hours, then plasma-clean to see if the oxide is removable without adding surface contamination.
- 2) “Seed” the surface of a clean split-cell with 10 and 0.3 μ Al<sub>2</sub>O<sub>3</sub> particles, one-half cell each.

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The seeding process involved mixing alumina polishing powder in pure water and depositing droplets of the mixture at specific areas on the cell surface. The droplet was dried in a nitrogen-flushed dry box, put into the scanning electron microscope (SEM) and imaged at the particle-seeding points. For accuracy of re-location, those areas were fiducialized prior to seeding using a diamond indenter.

### 3. Results

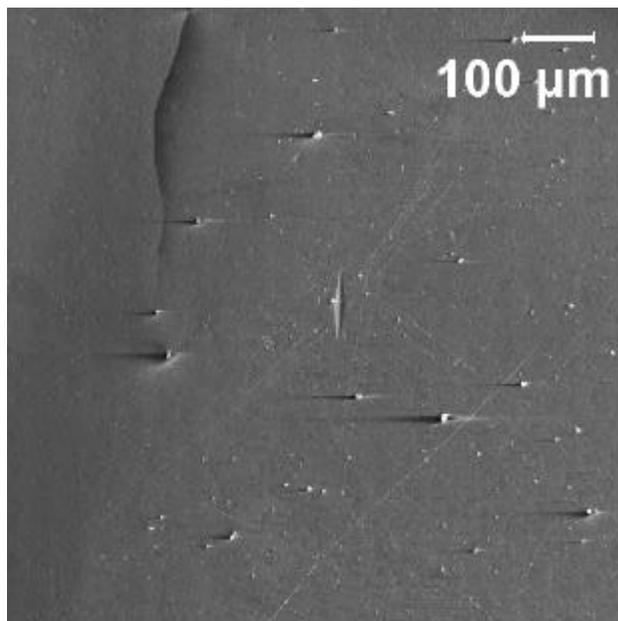
Table 1 shows the XPS results for the furnace-oxidized cells. Cell 107 is from mid-stack (three up from middle in stack of thirteen cells) and 107T is from the stack top, inside surface measured. These results can be compared to cells as they come from the Plating Shop, Cell 106, same table. The plasma process removes oxide without adding carbon contamination.

	Cu	O	C	Si	Cl	N
<b>107</b>	54	23	22	-	1	-
<b>107T</b>	59	23	17	-	1	-
<b>106</b>	37	33	23	2	1	4

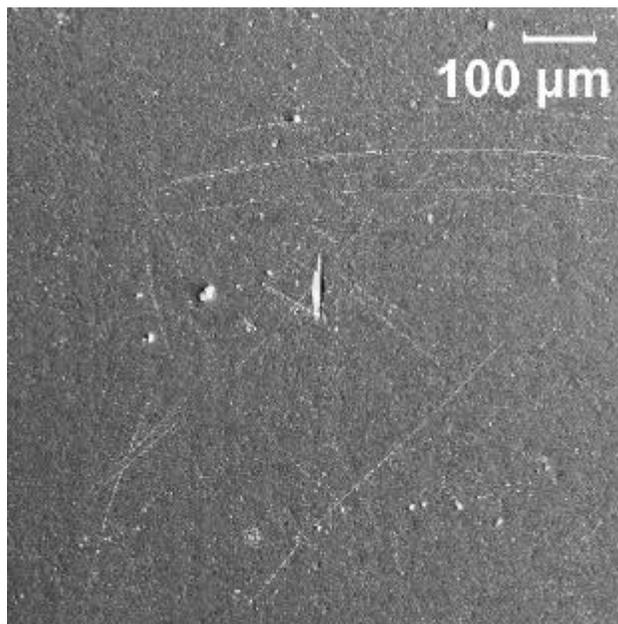
**Table 1. XPS atomic percentages of elements on Cu, before (106) and after (107,107T) plasma cleaning.**

Optically, the thermally oxidized surface before plasma processing shows interference colors of copper oxide. After plasma, the cavity area of the cell, penetrated by the plasma, is copper-colored but matte in finish. This is the expected case for a heavily-oxidized surface. It is the oxide that damages the surface finish. The plasma has no significant momentum-transfer ability to move metal. It merely reveals the pre-existing damage.

Figures 1-4 are a series of before/after SEM images of the seeded-particle Cell 073 halves. Note the factor of ten difference in magnification necessary to reveal the smaller particles. Again, this cell was mounted mid-stack. As mentioned above, the plasma process reveals oxide roughness but, in addition to thermal oxide, this applies to rough surfaces caused by water (as opposed to furnace) oxidation as well. Many SEM images taken showed a very random pattern of post-plasma particle redistribution. In some cases, the numbers of particles even rose in some areas.

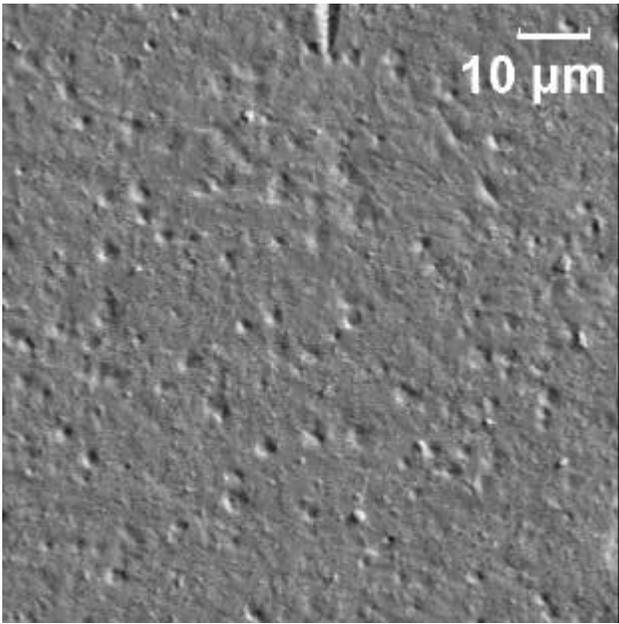


**Figure 1. Ten-micron particle-seeded surface, before plasma cleaning, 90° from slit-sawn edge (073EV-1).**

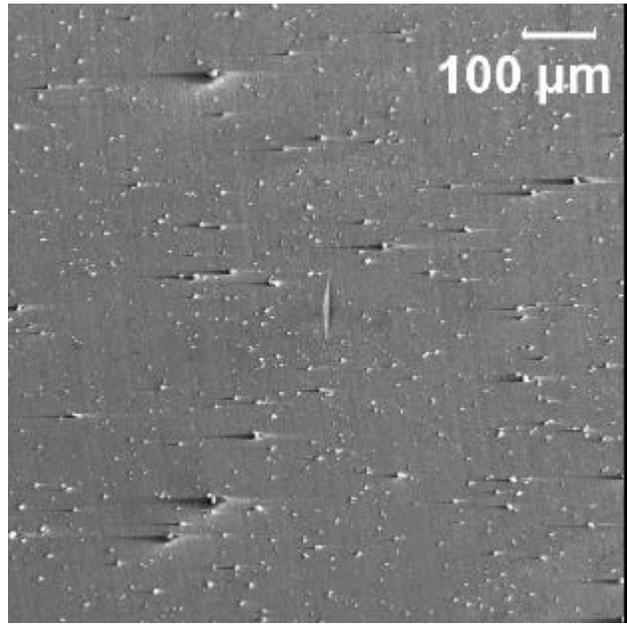


**Figure 2. Ten-micron particle-seeded surface, after plasma cleaning, 90° from slit-sawn edge (File #073EV-1a).**

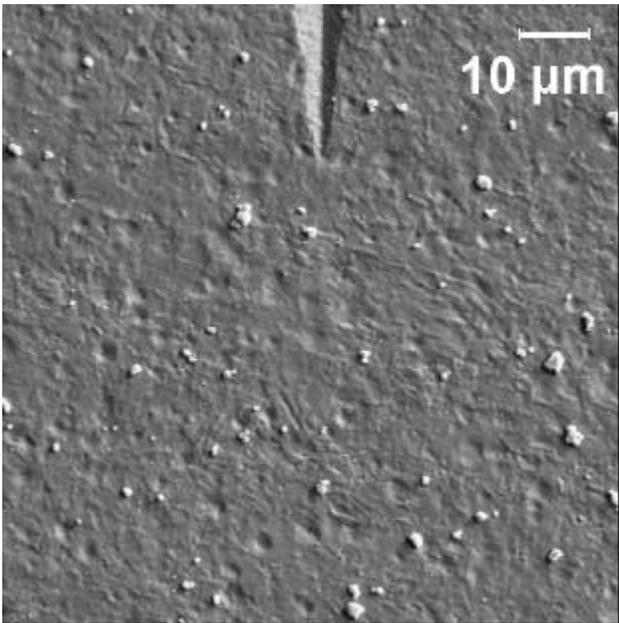
An automated SEM post-plasma search for particle identity showed a large number of silicon-based (presumably silicon oxide) particles. This must come from other areas of the plasma chamber; apparently, the plasma process stirs up loose material around the chamber and carries it to the interior surfaces.



**Figure 3.** 0.3-micron particle-seeded surface, before plasma cleaning, 90° from slit-sawn edge (073SV-1).

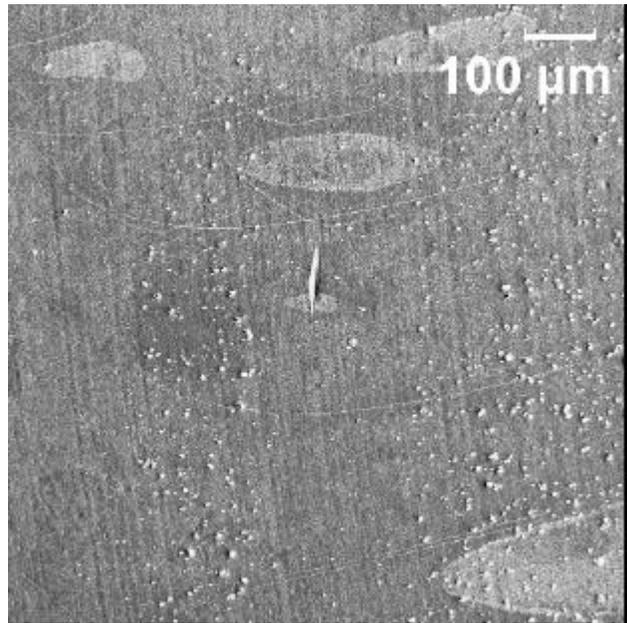


**Figure 5.** Ten-micron particle-seeded surface, before plasma cleaning and gap appearance of Figure 6, near slit-sawn edge (073EL-3).



**Figure 4.** 0.3-micron particle-seeded surface, after plasma cleaning, 90° from slit-sawn edge (073SV-1a).

Even stranger happenings were observed, as shown in Figures 5 and 6. Figure 6 show gaps appearing between uniform groupings of particles (for which we have no explanation).



**Figure 6.** Ten-micron particle-seeded surface, after plasma cleaning, same location as Figure 5 (073EL-3a). Shows strange development in density distribution and droplet appearance compared to Figure 5 image.

Atomic force microscopy (AFM) measurements on water droplet/no droplet areas show that this area is smoother in the water-oxidized area, following plasma. This is expected because the water oxide is thicker than the air oxide. Plasma will chemically-etch away the surface only until oxide is removed. The peak tops of the surface

roughness are most exposed and get etched preferentially. More etching in the water droplet area means a smoother surface. The numbers for average roughness,  $R_a$ , are 156 and 141nm for non-droplet and droplet areas, respectively with the AFM.

## 5. Conclusions

- 1) Particles from around the chamber get re-distributed by the plasma discharge. This phenomenon is well-known in the semiconductor business. They make provisions for actively sweeping particles away in their plasma systems.
- 2) Small and large particles are mixed together by the plasma process.
- 3) Removal of heavy surface oxide by the plasma results in a roughening in the previously thermal oxide-covered area.
- 4) The distribution of particles after plasma-cleaning is extremely non-uniform – some areas gain, while others lose, particles.
- 5) Following plasma, particles sometimes concentrate onto the droplet areas, right up to droplet edges.
- 6) New features appear (are revealed, likely) by the plasma process, including micro-drops, scratches and surface defects.

The next step in the plasma-cleaning development is to arrange for effective flow-through of gas from the system so that plasma-removed particles can be swept permanently out. Following that would be a study of the effectiveness of plasma reaching into the internal corners of the accelerating cavities so that particles are not simply “swept into the corner” when loosened by the plasma-processing on the irises.