PROCESSING OF O.F.E COPPER BEAM CHAMBERS FOR PEP-II HIGH ENERGY RING

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Using laboratory scale and full size PEP-II vacuum chambers, chemical cleaning, glow discharge and thermal process effects were evaluated using surface analysis by x-ray photoelectron spectroscopy (XPS). These processes were optimized to reduce surface carbon and thereby minimize photodesorption gas loads. The relation of surface carbon to ion dose was investigated and compared for pure argon, 5% oxygen in argon, and pure hydrogen plasmas. Argon incorporation was noted only when the copper was oxidized in the mixed gas. Surfaces, stable in ambient atmosphere, were obtained having surface carbon values less than 10%. These optimized recipes will be used in processing copper vacuum chambers for the PEP-II B-Facility.

I. INTRODUCTION

In electron storage rings, the main gas burden is due to synchrotron radiation desorption. Initial storage times are short until the intercepting walls can be “scrubbed” and the photodesorption yield (τ) can be reduced by orders of magnitude. Acceptable τ is usually not achieved until photon doses > 10^{11} photons/cm^{2} have been reached. Of course if the surfaces are repopulated when the beam is off for extended periods, leaks occur, or the system is vented, the surfaces must again be “scrubbed” to low values of τ.

Many researchers have studied methods of preparing beam chamber surfaces with reduced initial scrubbed desorption yields. (1-9) The surface processing techniques include controlled atmosphere extrusion and machining, wet chemical surface removal, electropolishing, ozone and oxygen purging, and many combinations of gas discharge plasma cleaning. To date, most of these studies have concentrated on alloys of aluminum and stainless steel whose surface oxides are relatively stable. Recently copper surfaces have begun to be investigated (10)(11)(12). This report outlines the work at SLAC on preparing OFE copper accelerator and klystron parts over a period of five decades. In order to minimize the final concentration of surface carbon, we modified this evolved cleaning recipe.

1. Steam clean
2. Alkaline soak in Enbond™ Q527 for 5 min @ 180°C
3. Cold tap water rinse for 2 minutes
4. Dip in 50% hydrochloric acid at room temperature
5. Cold tap water rinse for 2 minutes
6. Etch in acid solution (4 vol. %HNO3, 10 vol. %C2H4O2, 100 g/I CrO3, 5 ml/l HCl) for 5 minutes.
7. Cold tap water rinse for 2 minutes
8. Dip in hydrochloric acid at room temperature
9. Cold tap water rinse for 2 minutes
10. Cold de ionized water rinse (>1 megohm)
11. Cold de ionized water rinse (>6 megohms)
12. Hot (106°F) de ionized water rinse (>10 megohms)

II. TESTING TECHNIQUE

XPS was used to measure changes in surface composition on 5 cm² round discs cut from a beam tube extrusion. Surface composition changes were monitored through the wet cleaning process as bath compositions and rinse water conductivity were adjusted. These coupons were also inserted into a test chamber (Figure 1) to monitor changes in surface composition following glow discharge processing. Copper discs were also installed at three locations in full-scale dipole and quadrupole beam chambers to monitor thermal and glow discharge processing effects on surface composition (Figure 2).

III. OPTIMIZED COPPER CLEANING RECIPE

Stanford had evolved a recipe for cleaning copper accelerator and klystron parts over a period of five decades. In order to minimize the final concentration of surface carbon, we modified this evolving cleaning recipe.

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Note that the final rinse uses the lowest conductivity de-ionized water that is practical. If the resistivity of the final rinse water is less than 10 megohms, there is more residual surface carbon on the copper. Figure 3 shows some typical XPS surface composition results for the processes investigated.

### Surface Treatment | XPS Surface Atom %
--- | ---
Chem cleaned (old recipe) | Cu 22.4  O 22.5  N 11.9  Ar 1.6
Chem cleaned (optimized) | Cu 43.4  O 36.8  N 17.9  Ar 1.9
GDC (2e19 ions/cm²) | Cu 50.6  O 40.0  N 8.0  Ar 1.4
5% O₂-Ar in test chamber | Cu 48.6  O 42.0  N 8.0  Ar 1.4
GDC (2e18 ions/cm²) | Cu 64.2  O 23.6  N 12.2  Ar 1.4
5% O₂-Ar in beam chamber | Cu 50.6  O 40.0  N 8.0  Ar 1.4
GDC (2e18 ions/cm²) | Cu 64.2  O 23.6  N 12.2  Ar 1.4
Pure H₂ in test chamber | Cu 64.2  O 23.6  N 12.2  Ar 1.4

Figure 3. Table showing Copper Coupon Surface Composition (by XPS) for Various Processes

### IV. GLOW DISCHARGE PROCESSING FOR CARBON REDUCTION

Glow discharge processing, following chemical cleaning, can reduce surface carbon levels to less than 10 percent. Figure 4 shows the XPS carbon concentration data comparing the results obtained for pure argon and 5% oxygen-argon. Data points from the test chamber and the beam chambers fall on the same curves. The 5% oxygen-argon is more efficient than pure argon in removing carbon due to the combined chemical activity of the oxygen and the physical sputtering of the argon.

There may be a serious drawback to using oxygen with argon. With pure argon, the XPS results showed no argon on the copper surfaces, in either the test chamber or the beam chambers. However, all samples run in the beam chambers with the 5% O₂-Ar mixture show ~1% argon incorporation. Mathewson (13) also found argon burial when using O₂-Ar. None of the copper discs run in the test chamber with 5% oxygen-argon showed argon incorporation. The ion flux in the test chamber was about a factor of ten higher than in the beam chamber and the sample temperature was higher, resulting in some visible oxidation on the surface.
of formation nearer -38 Kcal/mol and therefore can be
easily disassociated at low energy and easily reduced with
hydrogen.

Pure H\textsubscript{2} plasma processing of copper has many
advantages over argon-oxygen, one of which is very
limited sputtering (14). Sputtering may produce micro
particles which have been postulated to result in stored
beam degradation.

One limitation to using H\textsubscript{2} discharges to clean the PEP-
II dipole chambers is that the H\textsubscript{2} plasma must be employed
prior to installing the distributed ion pumps to avoid
loading the pump titanium with hydrogen that might not all
be removed by baking at 200°C.

V. HYDROGEN DISCHARGE PROCESSING

In view of the above considerations with regard to
oxidation and argon incorporation when using the mixed
gas plasma, there may be some incentive to use a pure
hydrogen plasma for removing carbon and oxygen from
OFE copper surfaces. XPS coupons discharged in pure
hydrogen in the test chamber showed somewhat faster
carbon reduction than those discharged in 5% oxygen-
argon. Figure 5 compares the XPS spectra of coupons ion
bombarded to the same dose (2x10\textsuperscript{18} ions/cm\textsuperscript{2}) with
5% oxygen-argon in a quadrupole chamber (A) and run in the
test chamber in a pure hydrogen plasma (B).

![XPS Spectra of copper samples GDC processed in 5% O\textsubscript{2}-Ar (upper) or H\textsubscript{2} (lower) to 2x10\textsuperscript{18} ions/cm\textsuperscript{2}.]

VI. CONCLUSION

We have shown that it is possible to clean OFE copper
surfaces chemically, to yield surface carbon levels near
20%. These levels can be further reduced to about 10% by
glow discharge cleaning with argon, oxygen-argon, or
hydrogen plasma discharges. The cleaned, baked and
discharged surfaces can be stored in clean air for weeks
with little change in surface composition as measured by
XPS.

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