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

E. Hoyt, M. Hoyt, R. Kirby, C. Perkins, D. Wright, A. Farvid, Stanford Linear Accelerator Center, Stanford University, Stanford, CA 94309 USA

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-Factory.

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 (η) can be reduced by orders of magnitude. Acceptable η is usually not achieved until photon doses > 10^{23} 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 η.

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 beam chambers for the PEP-II B Factory. We focused on processes that yield minimum residual carbon on the surface, reasoning that the initial and ultimate CO and CO_2 photodesorption yields would be correspondingly lower.

II. TESTING TECHNIQUE

XPS was used to measure changes in surface composition on 5 cm^2 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).

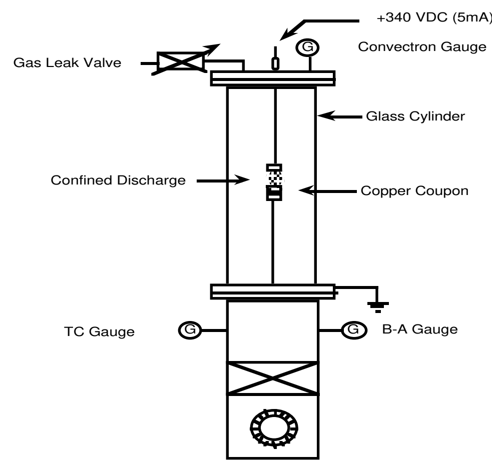

Figure 1. Experimental setup for testing copper discs

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 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/l 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)
13. Blow dry with nitrogen gas.

[*] Work supported by U. S. Department of Energy, contract number DE-AC03-76F00515

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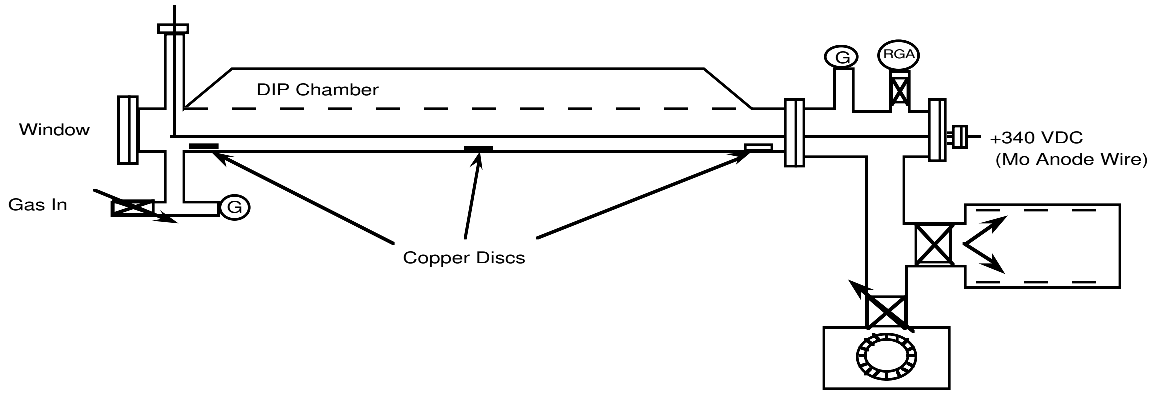

Figure 2. Schematic Diagram of Glow Discharge Set-Up for Dipole Vacuum Chamber Processing Showing XPS Coupon Location

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 %*					
	Cu	*O*	*N*	*C*	*Cl*	*Ar*
Chem cleaned (old recipe)	*22.4*	*22.5*	*11.9*	*41.6*	*1.6*	-
Chem cleaned (optimized)	*43.4*	*36.8*	-	*17.9*	*1.9*	-
GDC (2e19 ions/cm^2) 5% O_2-Ar in beam chamber	*50.6*	*40.0*	-	*8.0*	-	*1.4*
GDC (2e18 ions/cm^2) 5% O_2-Ar in beam chamber	*48.6*	*42.0*	-	*8.0*	-	*1.4*
GDC (2e18 ions/cm^2) Pure H_2 in test chamber	*64.2*	*23.6*	-	*12.2*	-	-

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% 0_2-Ar mixture show ~ 1% argon incorporation. Mathewson (13) also found argon burial when using O_2-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.

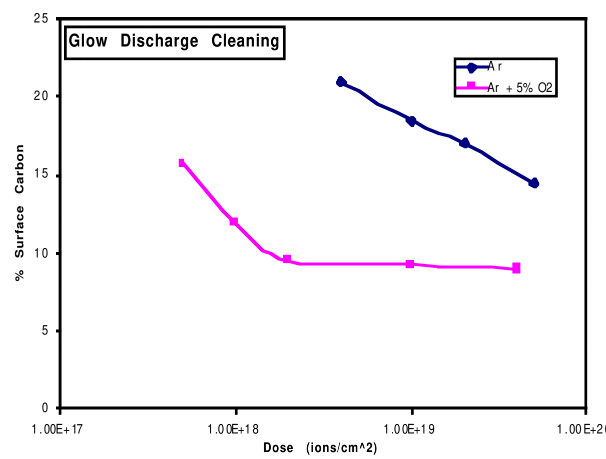

Figure 4. Surface carbon levels versus ion dose for pure argon and 5% oxygen-argon discharges

There is a definite increase in the surface oxygen levels following processing with 5% 0_2-Ar. Argon may be incorporated or buried in the growing oxide depending on the temperature. If it were possible to discharge, with oxygen-argon, at a high enough temperature to preclude oxide growth, argon might not get occluded. These are subtle effects that may need study. Dylla's very comprehensive review article on GDC (14) deals principally with stainless steel surfaces, but sheds no light on copper. Some work has also been reported on aluminum alloy discharge cleaning. Copper surfaces are quite different. For example, Cr_20_3 has a free energy of formation of -142 Kcal/mol and Al_20_3 has a free energy of formation of -408 Kcal/mol whereas Cu_20 has a free energy of formation nearer -38 Kcal/mol and therefore can be easily disassociated at low energy and easily reduced with hydrogen.

Pure H_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_2 discharges to clean the PEP-II dipole chambers is that the H_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 ($2x10^{18}$ ions/cm^2) with 5% oxygen-argon in a quadrupole chamber (A) and run in the test chamber in a pure hydrogen plasma (B).

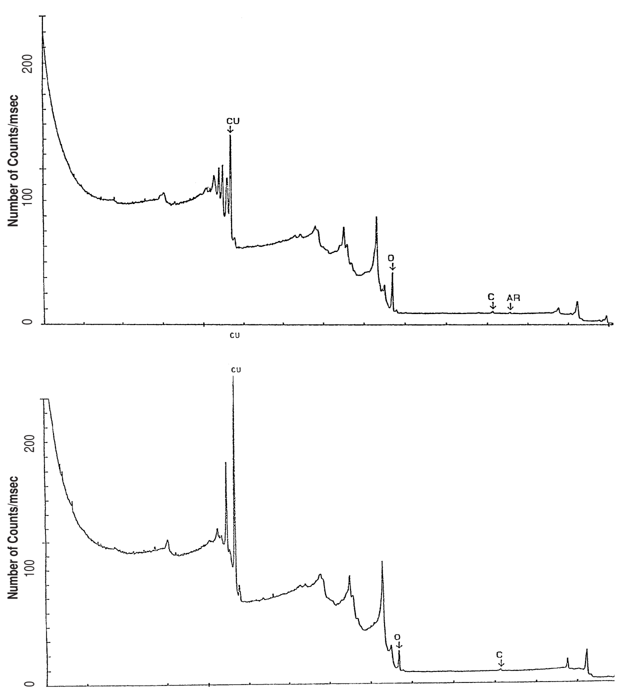

Figure 5. XPS Spectra of copper samples GDC processed in 5% O_2-Ar (upper) or H_2 (lower) to $2x10^{18}$ ions/cm^2 .

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.

VII. ACKNOWLEDGMENTS

W. Stoeffl of LLNL provided much advice and Rose Darrough assisted with the beam chamber discharge and outgassing tests. Thanks to Ed and Nadine for their late-night formatting help.

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