

QUANTUM EFFICIENCY AND TOPOGRAPHY OF HEATED AND PLASMA-CLEANED COPPER PHOTOCATHODE SURFACES*

Dennis T. Palmer, Titan PSD, San Leandro CA 94577, U.S.A.
R.E Kirby and F.K. King, SLAC, Menlo Park CA 94025, U.S.A.

Abstract

We present measurements of photoemission quantum efficiency (QE) for copper photocathodes heated and cleaned by low energy argon and hydrogen ion plasma. The QE and surface roughness parameters were measured before and after processing and surface chemical composition was tracked *in-situ* with x-ray photoelectron spectroscopy (XPS). Thermal annealing at 230°C was sufficient to improve the QE by 3-4 orders of magnitude, depending on the initial QE. Exposure to residual gas slowly reduced the QE but it was easily restored by argon ion cleaning for a few minutes. XPS showed that the annealing or ion bombardment removed surface water and hydrocarbons.

INTRODUCTION

Quantum efficiency (QE), defined as photo-emitted electrons per incident photon, measurements often show a variation across a copper cathode surface, which is usually interpreted as caused by adsorbed contamination [1]. We are interested in determining the effect of typical bakeout conditions (230°C) on the QE, and how that result compares to Ar plasma ion sputtering (previously used to remove contaminants [2]).

EXPERIMENTAL SETUP

The QE measurement/loadlock and x-ray photoelectron spectroscopy (XPS) chambers are separated by a gate valve. A schematic diagram of the XPS system along with the load lock system used for our QE measurements is shown in Figure 1. Plasma cleaning, using either argon or hydrogen, was conducted in the QE chamber. Annealing of the samples was done in the XPS chamber, using a heating stage-equipped manipulator.

Copper samples were manufactured from ASTM-standard OFE class one copper. The 1" diameter polycrystalline copper samples were first chemically-etched in the SLAC Plating Shop using the PEP-II high energy ring recipe [3]. The samples were then polished and ultrasonically-degreased, following standard metallographic procedures.

QE Measurement

The load lock chamber incorporates a plasma source and sample transfer platen for moving the sample into the

XPS chamber. The sample was mounted onto an insulated sample holder on the transfer platen. The sample was illuminated, through a deep UV-grade vacuum window, using a xenon lamp and monochromator. Photoemission currents were monitored by connecting the sample to a shielded feedthrough, external bias battery and electrometer. The sample bias was -18V, used to prevent photoemitted electrons, accidentally generated at the chamber walls, from reaching the sample.

UV Light Source

The wavelength accuracy of the scanning monochromator is ± 2 nm. The light spot size on the copper samples was 0.15 x 0.20 inches. The spot asymmetry is due to the non-normal 40° incidence angle of the UV light source with respect to the sample normal. A nitrogen gas purge was used to eliminate water-vapor UV attenuation in the light source, monochromator, and transport tube prior to its passage through the UV-grade window. The UV light intensity was measured with a calibrated UV photodiode placed, at the sample location, in the load lock with a nitrogen atmosphere. The intensity versus wavelength data was incorporated into the QE DAQ system and used to correct collected data as a function of wavelength.

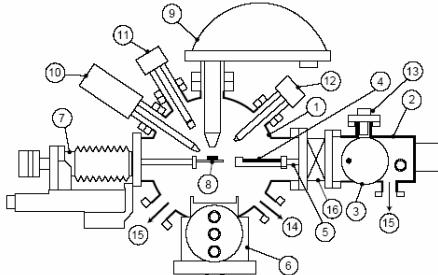
Plasma Source

The low energy ion source (Ion Tech, Ltd., model B-15) is attached to the load lock chamber and impinges the sample at a 40° from sample normal. The ion beam divergence is approximately 9° with an gun aperture size of 1.5 mm. The ion impingement spot is elliptical, 15 x 20 mm. The ion source is capable of producing ions with energies from 100 eV to 3650 eV. Argon or hydrogen gas is injected directly into the ion source through a capillary. Pressure in the load lock rises to a few mTorr during plasma cleaning. Base pressure, prior to measurements and processing in the cryogenically-pumped load lock, was 1×10^{-8} Torr.

XPS Chamber

X-ray photoelectron spectroscopy measures the chemical composition of the top five nanometers of surface. Soft x-rays stimulate the ejection of photoelectrons whose kinetic energy is measured by an electrostatic energy analyzer (VG Escalab II). Small changes to the emitted photoelectron energy are caused by chemically-shifted orbitals from which the electrons are ejected; thus, the measurement provides chemical state information about the sample surface. XPS data is collected from a 4 mm diameter area on the sample, within the area bombarded by the plasma source.

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XPS/SEY/SEM/AES/EDX/LEXRF/PES System

(1) Analysis chamber	(9) Electrostatic energy analyzer
(2) Loadlock chamber	(10) X-ray source
(3) Sample plate entry	(11) SEY/SEM electron gun
(4) Sample transfer plate	(12) Micro focus ion gun
(5) Rack and pinion travel	(13) Sputter ion gun
(6) Sample plate stage	(14) To pressure gauges and RGA
(7) XYZΦ Omniax™ manipulator	(15) To vacuum pumps
(8) Sample on XYZΦ	(16) Gate valve

Figure 1: Experimental system, showing load lock and XPS chambers.

Topography Measurements

Surface topography was measured with a Burleigh Inc. Metris atmospheric atomic force microscope (AFM), in silicon probe contact mode, with a tip radius of 15 nm or so, depending on wear. AFM calibration was monitored by periodic measurements of x, y and z gratings from NT-MDT Co.

RESULTS

Two polycrystalline copper samples were cleaned and polished as described in Experimental Setup. The first sample studied, Master_0, had an initially-disappointing QE (Figure 2). The sample then underwent a thermal annealing for approximately 1.5 hours at $T_{max}=230^{\circ}\text{C}$, in the XPS chamber. This resulted in a QE increase of over three orders of magnitude at 265 nm.

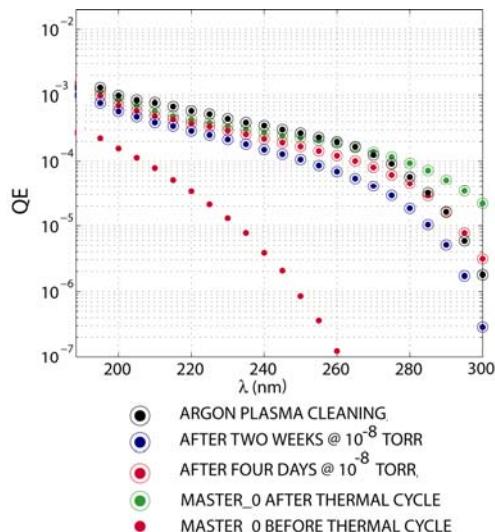


Figure 2: Quantum efficiency data from sample Master_0, corrected for UV intensity vs. wavelength.

The Master_0 sample was left in a vacuum (95% H₂, 5% CO) of 10^{-10} Torr for 4 days, in the XPS chamber. The resulting QE at 265 nm dropped from 1.6×10^{-4} to 1.0×10^{-4} . After 14 days further exposure, the QE drifted down to 5.4×10^{-5} . Plasma cleaning with 1 keV argon ions, at an average current of 15 μA , restored the QE to 1.7×10^{-4} .

The second copper sample, PCC-1, had an even more disappointing initial QE than Master_0 (Figure 3). PCC-1 was then annealed for 3 hours at a $T_{max}=230^{\circ}\text{C}$. The resulting QE was then similar to that of Master_0 after annealing.

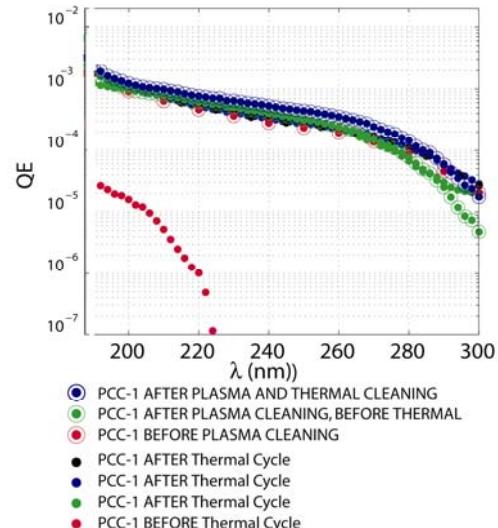


Figure 3: Quantum efficiency data from sample PCC-1, corrected for UV intensity vs. wavelength.

XPS Chemistry

XPS scans were conducted on Master_0 and PCC-1 before, during, and after thermal bakeout and after 1 keV ion plasma cleaning. Figure 4 shows representative XPS data, this particular data after argon ion cleaning. Table 1 lists the surface atom% concentration of the elements present within the top 5 nm or so of surface.

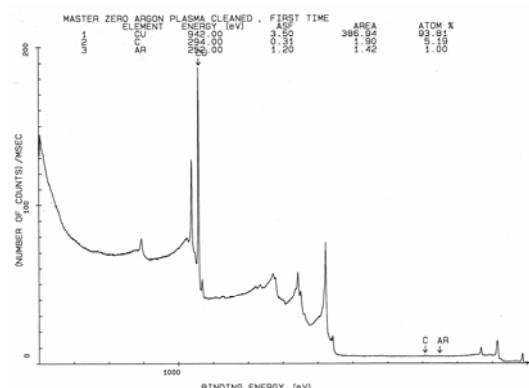


Figure 4: Master_0 XPS spectrum, after argon ion cleaning.

Removal of surface oxygen (present mostly as adsorbed water) and hydrocarbons (tracked through chemical shifts) accounted for the largest increase in QE. After the plasma treatment, only a small (relatively!) further improvement was seen in QE, compared to the dramatic change following annealing; however, the copper oxide and surface amorphous carbon were essentially removed. This clearly suggests that water and hydrocarbons reduced the initial QE.

Element	Initial	Annealed	Residual Gas	Ar ions
Copper	28.3	55.6	45.6	93.8
Carbon	41.2	29.2	38.6	5.2
Argon	-	-	-	1.0
Oxygen	28.1	9.7	12.9	-
Sodium	2.0	4.6	1.7	-
Calcium	0.4	0.9	1.2	-

Table 1: Surface atomic% concentration on Master_0, after each processing step.

PCC-1 was processed somewhat differently, with hydrogen ions replacing argon and a second annealing step after hydrogen plasma. Table 2 lists the PCC-1 results.

The initially-high carbon level explains the lower-than-Master_0 QE. The hydrogen plasma was not as successful as argon at removing remaining contaminants following the annealing step. However, the sample mirror finish was removed by the heavier argon ion bombardment on Master_0 but PCC-1 was unaffected, at least visually, by the much lighter hydrogen ions.

Element	Initial	Annealed	H ions	Re-annealed
Copper	14.1	53.6	76.9	70.8
Carbon	50.8	26.1	6.5	13.3
Oxygen	31.3	14.1	8.7	6.8
Sodium	3.8	6.2	7.9	8.5
Calcium	-	-	-	0.6

Table 2: Surface atomic% concentration on PCC-1, after each processing step. XPS does not detect hydrogen.

AFM Topography

The AFM measurements were conducted on both Master_0 and PCC-1, as polished, and after vacuum thermal and plasma processing. The results are presented in Table 3. Only small increases in both Ra (average roughness) and Rq (peak-to-peak roughness) resulted from processing. The loss of mirror finish on Master_0, caused by the argon bombardment and apparent by eye, does not appear “reflected” in the Ra result.

	As Polished		After Processing	
	R _a (nm)	R _q (nm)	R _a (nm)	R _q (nm)
Master_0	12.3	17.2	15.8	20.1
PCC-1	14.0	17.9	20.7	25.8

Table 3: AFM data for Master_0 and PCC-1 before and after processing.

CONCLUSIONS

We have shown that over three orders of magnitude improvement in the quantum efficiency (QE) can be expected due to the application of rf gun-type bakeout temperature heating. This is due to the removal of surface adsorbed water and hydrocarbons. In addition, a factor of two increase in the QE was seen due to argon plasma cleaning. No significant topography changes were noted by AFM, following heating and sputtering, but argon ion bombardment caused a visible change in surface reflectivity.

Previous work using Ar plasma cleaning of copper achieved a factor of two improvement in QE, at higher photon energy [2]. Carbon and oxygen-containing contaminants were identified in that study from k-edge absorption measurements, consistent with our identification of water and hydrocarbons. Argon bombardment does damage the surface, however, making attractive the cathode heating alternative, provided that the cathode is kept as the hottest part of the vacuum system and does not simply become a getter pump for contaminants desorbed from neighboring surfaces. Hydrogen plasma is an attractive alternative for cathode re-cleanings, in that surface damage is not an issue plus the danger of opening vacuum leaks, as a result of multiple bakeouts, is eliminated.

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