Common Sense Copper and RF Guns

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A number of properties and treatments of copper relevant to the construction and operation of RF photoemission guns is archived for common reference by the LCLS collaboration.

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Abstract

The purpose of this document is to gather together both fundamental information on copper and on the cleaning and operation of copper in RF gun structures. While incomplete, this is a living document and will be added to and updated as necessary.

Introduction

General notes on copper

Copper (Cu) is a face-centered cubic (fcc) metal noted for its malleability and high thermal and electrical conductivity[1]. It is this latter property which makes it particularly useful in radio frequency devices. The electronic configuration of copper is $[\text{Ar}]3d^{10}4s^1$ which means that most of the conduction is via the s electrons as the d electrons are located too far below the Fermi level to contribute significantly. Copper has a relatively low melting point of 1358K but is surprisingly heavy with a density greater than steel.

Figure 1. The fcc structure. Only those faces that can be seen in this orientation have the in-face atoms shown. The cubic cell of copper has $a = 3.61\text{\AA}$.

The skin depth of copper as a function of frequency is shown below in figure 2. From this plot it is clear that the critical surface selvage is of the order of 10 microns. In other words, the driving fields of a typical S-band RF gun do not penetrate with much amplitude beyond this depth. It is over this distance that the cavity material must be of highest quality. In addition to the obvious reason that the surface currents must be able to flow with minimal resistive losses, the low defect density is necessary to prevent RF field induced breakdown.

Figure 2. Skin depth of copper as a function of frequency[2].

Macroscopic form

Copper can be produced in single crystal or polycrystal forms. The polycrystal form is not to be

confused with amorphous copper which is an entirely different material. The primary differences between single crystal and polycrystal copper are the size and relative orientation of the microcrystals, or grains. In single crystal copper, the grains have all their axes nearly parallel and the boundaries between the grains are small and infrequent. The quality of a single crystal material is specified by its mosaic spread, a measure of the non-parallelness of the microstructure. The production of single crystal copper is limited to specialized applications and the handling of the material must be done with care as stress tends to cause multifaceting.

Polycrystal copper is far more common and is the form encountered in everyday applications. The size of the grains is influenced by every form of mechanical and thermal process in the material's history up to remelting. High temperature anneals must be performed with care lest undesirable grain growth occur. The volume between grains, or grain boundaries, is comprised of some number of defect sites. A defect refers to any deviation from the perfect crystalline state. Defects can occur in the form of absent atoms (vacancies or voids), displaced atoms (interstitials) and elementally different atoms (contaminants). Due to the large number of empty sites, grain boundaries are ideal sinks for diffusive species, such as gas atoms. The hardness, malleability and thermal and electrical conductivities are all dependent on the grain structure.

Grades of polycrystalline copper

The grades of copper may be divided in a number of fashions. The first is to differentiate between material that has fire or electrolytically refined from the original blister copper. Electrolytic refining refers to the purification of refined copper via an electroplating technique. Final alloy grading is through the UNS number or its equivalent, a listing of the UNS numbers can be found in ref. [3]. Another post-refining step known as hot isostatic pressing (HIP) may be applied. This process includes exposure to high temperature and an isostatic gas for pressurization, typically argon. Through a combination of plastic deformation, creep and diffusion, many internal voids and some microporosity is eliminated. HIP-ing copper does not significantly change its chemical composition (see below).

Oxygen free copper should not be confused with de-oxidized copper which is copper that has had a small quantity of a de-oxidizer, typically phosphorous, added to it. Oxygen free copper is made through the process of remelting and pouring in a carbon monoxide and nitrogen atmosphere[4].

Copper is further classified through the microscopic examination of its structure. Important details of the structure include the grain size and the density of defects. A set of standards for structure comparison is available from the American Society for Testing and Materials[5].

Limitations of copper purity standard

The ASTM limits on sulphur contamination for OFE copper is 18 ppm. It is possible to derive the worst case effects from this limit as follows. The emission sites mapped by field emission microscopy [Pupeter et al.] show a propensity toward sulphur as a primary constituent with the emitters having a median diameter of 0.5 micron. In this illustration we take a 1 cm³ volume of copper as shown in figure 3 and calculate the probable emission site density based on very pessimistic assumptions.

Figure 3. Raisin bread model showing copper cube with size exaggerated spherical sulphur contaminant sites.

First, we assume that the sulphur will aggregate into spherical contaminant sites with a diameter of 0.5 micron. From the allowed concentration, we expect up to 18×10^{-6} of volume to be sulphur atoms (assuming equal atomic volumes of sulphur clusters and copper metal), or $1.8 \times 10^{-5} \text{ cm}^3$ is occupied by sulphur atoms. Taking the cluster diameter to be 0.5 micron, and hence an individual cluster volume of $6.54 \times 10^{-14} \text{ cm}^3$, this yields a net of 2.8×10^8 sulphur clusters per 1 cm^3 copper volume. Allowing clusters to be have maximum separation, each cluster 'owns' a copper volume of $3.57 \times 10^{-9} \text{ cm}^3$. Therefore, a 0.5 micron slice at the top of our copper cube has an expected occupancy of 1.4×10^4 sulphur clusters. Now one very unreasonable assumption has been made, in that sulphur will not generally aggregate as suggested; however, this serves to illustrate that the level of contamination permitted for this highest grade of copper may still be too high to ever achieve a surface free of field emission sites without additional bulk contaminant depletion. Furthermore, etching the surface without mechanical abrasion can lead to exposing emitting sites in addition to smoothing the copper surface.

Pre-installation material preparation

General cleaning notes

The objective in fabrication is to generate a part made from the highest purity copper that correctly mates with the receiving system and to do so in a fashion that leaves exposed only the best properties of the raw material. Except for elements that may be removed through a reducing or oxidation treatment, such as in a hydrogen furnace or during a high temperature bake, such as to achieve UHV conditions, the installed part will generally not be any cleaner than achieved in its last preparation stage. Exceptions to this rule are found in cases where an *in situ* cleaning method can be applied (see below). These methods are generally limited to cleaning the near surface region unless a method of bulk contaminant depletion is employed. We first begin by recounting some cleaning experiences.

Klystron breakdown studies

The Klystron department has been investigating a series of preparation methods to reduce breakdown on test pieces placed in a high RF field. Their experiences are:

Machining

With no special preparations beyond using a clean mill or lathe, 7 micron roughness surfaces are achieved. One prohibition is to not allow the use of abrasive pads[6] for cleanup of the part or the tool after work has begun. It was found that their use led to imbedded particles in the copper surface.

Cleaning

The test nose pieces are cleaned with etch procedure C1 slightly modified by omitting the oxyban etch stage. It was found that the etch rates, nominally 1 micron per minute, had a strong dependence on the sequence of samples; later samples etched slower. Weakened solutions were found to be the underlying cause, so fresh etchants were put in place as a requirement. Particle contamination from the cleaning steps were also found to be a problem, so a new low particle filtration system is in progress of being set up in the soft wall clean room next to the plating shop.

Handling

Aside from cleanliness, the particle contamination of the surface of the test pieces was found to be very sensitive to mode of transport. Good results were had in using immersion in SLAC standard cleaning alcohol in special transporter designed to hold the part inverted in the liquid. Parts in need of installation were only handled in a laminar flow hood. It was found necessary to keep the surface irrigated as the dry, exposed surface accumulated particles.

GTF

Only one cathode plate has been tested to date in the GTF gun. This plate was machined at UCLA with final polishing, TiN coating and brazing occurring at SLAC. No special effort was made in handling beyond maintaining chemical cleanliness. *In situ* cleaning was used to increase the photoyield over the as-baked value.

Photoyield sample

A sample used in the first low voltage, continuous emission photoyield tests for the LCLS photocathode research was cut from a Class 1 OFE polycrystalline plate with a strain-free

diamond blade. It was polished while mounted on a glass plate with aqueous-based diamond polishing compound down to 0.25 micron. Post polishing cleaning consisted of the procedure used with the GaAs cathodes, namely degreasing in trichloroethane and rinsing in methyl alcohol. This sample was installed using a loadlock (no baking) and with no additional cleaning had a yield of 2.3×10^{-4} at 266 nm as seen in Figure 4 below.

Figure 4. Polycrystalline copper photoyield. The sample was biased to overcome the work function difference between the copper and the surrounding vacuum chamber.

PEPII copper vacuum chambers

Preparation of the copper vacuum chambers for the high energy ring employed both a somewhat different chemical recipe than the C1 method with the addition of post-chemical treatment glow discharge cleaning[7]. Glow discharge cleaning works primarily through the chemical action of the liberated ions, although heavy ions and excessive accelerating potentials can lead to potentially undesirable sputtering. Good reduction in the residual surface carbon was achieved with either Ar-O₂ or H₂ as the reaction gas.

Brookhaven preparation method

The Brookhaven group has made a comparison of the yield from copper under different preparation conditions[8]. The greatest difference was found in the type of polishing material used. They used both a non-aqueous based compound made by Excel and an aqueous based compound from Buehler with the highest yield from material polished with the latter substance. Considering the relatively poor vacuum conditions, 10^{-8} to 10^{-9} Torr, under which the tests were performed, it is misguided to make an extrapolation to expected performance in a system possessing a better vacuum.

KEK tests

Surface quality was measured on HIP Class 1 copper as a function of process[9]. Lowest surface carbon was obtained on material that had as its final stage an electropolish while the oxygen content was enhanced over that obtained on material which experienced one form of an ultrapure water based rinse. All material was diamond machined to a 0.05 μ m finish. Field emission tests from electrodes made with the low carbon surface showed nA level emission for field strengths of up to 28 MV/m.

Clear courses in cleaning

While they may now seem obvious, restatement of the useful methods from above follows.

1. Use the best material possible, meaning Class I OFHC copper.
2. Machine the part under clean conditions taking care to use a non-etching lubricant such as alcohol
3. Do not bring the machined surface in contact with anything that may contaminate it either through chemical reaction or via particle embedding
4. Use clean, particle free chemicals in the final stages of cleaning process

5. Do not allow the part to dry out; particles trapped in fluid are more easily removed than those trapped on the part
6. Polishing compounds should not have anything in them that cannot be removed from the surface
7. Transport of the part should always be in either a fluid (preferable) or in a nonstatic transport.
8. The part should only be exposed to high purity air
9. If electropolishing is desired, it should be combined with some form of mechanical abrasion to eliminate the 'raisin bread' effect.
10. The part should proceed from its final cleaning when it is in its chemically cleanest state to installation as soon as practical.

Potential cleaning methods

Areas that need some further investigation include bulk contaminant depletion and surface chemical quality assessment following installation and bake, potentially in a mock setup.

Suggested tests and preparatory work

The procedures for obtaining a clean, nearly particle free surface are well understood. Implementation of the processes is a matter of some small effort; however, maintaining the surface quality upon installation into the RF gun is a large hurdle since the process of installing a new cathode plate currently involves baking the entire gun. For this reason it is necessary to undertake some assessment of post installation cleaning methods.

Post installation processes and caveats

In situ cleaning

It is clearly necessary to undertake some form of cleaning the copper surface post installation and bake. A variety of *in situ* surface cleaning methods exist and they can be classified under two headings; chemical reaction or material transport. A chemical reaction may be with a reactive species such as atomic hydrogen or mediated by electrons in a purely desorptive process. Material transport broadly includes sputtering, field and laser induced ablation and contaminant diffusion under RF, thermal radiation or laser promoted heating. The former may lead to a roughened surface while the latter can potentially contaminate the surface rather than clean it. For these reasons, such techniques should be approached with some degree of caution. A review of cleaning methods leading to atomically clean surfaces for a variety of elements can be found in ref. [10]. Below we describe some techniques which may be considered and discuss their utility and drawbacks.

Laser cleaning

Laser cleaning encompasses both classifications through contaminant photodesorption and surface melting or ablation. The effect which occurs depends on the adsorbates and the laser energy and time structure[11]. Beyond a critical value of irradiance, the surface can be melted which is not generally desirable. In the presence of a high electric field, the problems can be worsened by the formation of instabilities in the melt and formulation of droplets[12].

Effects of electron bombardment

Electrons are very effective mediators for molecular bond disassociation. Their energy is not, however, usually high enough to generate atomic displacements as seen in ion collisions (see below). While they may mediate the dissolution of a bond, such as an atom on a surface, they do not usually provide sufficient momentum to allow the atom to escape. However, they can crack gasses near the surface, for example CO and CO₂ from the ambient vacuum, leading to carbon deposition on the surface. For these reasons, ion beam are used instead in cleaning material surfaces.

Hydrogen cleaning

A form of chemically induced desorption is the use of atomic hydrogen which may be generated by chemical cracking of the hydrogen molecule or via plasma disassociation. The latter can also lead to some ion induced cleaning (see below) if the atoms are not slowed through nonrecombinative surface collisions. In principle, the molecule could also be cracked with an intense photon beam. Some thermal energy must be present to provide the necessary additional energy for desorption of the hydrogen-contaminant (oxygen or carbon) complexes. Of concern in composite systems is

the presence of materials that may be harmed through either atomic or molecular hydrogen interaction with the pressures these processes require. An example is the loading of titanium plates in ion pumps.

Argon ion or cleaning

A standard technique used for the generation of atomically clean copper surfaces involves the removal of contaminants with an impinging argon (or neon or other noble gas) beam with an energy ranging from a few 100 eV to several kV. A surface that has been treated in such a fashion becomes disordered since copper atoms will be displaced as are the contaminant atoms. Reorganization of the local crystalline lattice involves the application of heat to increase the mobility of the copper atoms which then seek their lowest energy state, the well organized crystalline structure. During the heating process, new contaminants can diffuse to the surface, so many cycles of this process are usually necessary to deplete the near-surface region[13] of contaminants.

RF assisted laser cleaning

The application of high strength fields can lead to field induced desorption. If a surface is already heated, such as with an intense laser beam, then the process of field-assisted thermal desorption can take place. This is a highly localized process due to the intense laser pulse needed for high temperatures. It is very easy for such a process to lead to extreme variations in surface quality and composition over small areas.

Recommendations

Until a reliable cleaning process has been proven, tests on preparation should be conducted in a system that is not an RF gun, but a system where samples can be rapidly subjected to test processes and then analyzed. Ideally, some form of sample diagnostic should be implemented in any new RF gun design through the inclusion of additional cathode line-of-sight ports.

Other Effects

Copper particles migrating under the action of high fields

It is certainly insufficient to clean just a cathode plate if full dark current suppression is desired. Migration can occur any time the gun cavity is vented from laminar flow propelled particles. Additionally, particles can move due to the action of electric fields; 1 to 10 micron particles have been observed to migrate under a field of 0.5MV/m with a voltage spike of 10 ms duration[14].

Work function change induced by deformation

Nonuniformity of the cathode plate work function and consequently the yield can be further exacerbated by the addition of mechanical stresses. We can represent the force of the tuning nut on the rear of the GTF gun by the following drawings. The two thicknesses are considered since the cathode plate is of variable thickness. The different thicknesses correspond to the extremes of the plate material.

The deformation is given by[15]:

$$w = \frac{P}{8\pi D} \left[-(x^2 + b^2) \ln \frac{a}{b} + (x^2 - b^2) + \frac{1}{2} \left(1 + \frac{b^2}{a^2} \right) (a^2 - x^2) \right],$$

for $(x < b)$ and

$$w = \frac{P}{8\pi D} \left[-(x^2 + b^2) \ln \frac{a}{x} + \frac{1}{2} \left(1 + \frac{b^2}{a^2} \right) (a^2 - x^2) \right]$$

for $(x > b)$, where P is the applied force and

$$D = \frac{Eh^3}{12(1 - \mu^2)},$$

where E is the modulus of elasticity and μ is Poisson's ratio. For copper, $E = 16 \times 10^6$ lbs/in², $\mu = 0.340$ and the GTF cathode plate has $a = 1.5$ " and $b = 0.5$ " and $h =$ either 0.138" or 0.430". With a maximum deformation of 0.005", the required pressure from above is 7.3×10^3 lbs for $h = 0.138$ " and 2.7×10^4 lbs for $h = 0.430$ ". However, taking the force as equally distributed over the diameter of the nut, the maximum stress is located at the center of the cathode and is given by

$$\sigma_{x_{\max}} = \frac{3}{2} (1 + \mu) \frac{P}{\pi h^2} \left(\ln \left\{ \frac{a}{b} \right\} + \frac{b^2}{4a^2} \right),$$

such that

$$\sigma_{x_{\max}} \approx 2 \times 10^5 \text{ lbs/in}^2 \approx 10^3 \text{ MPa}.$$

The dependence of work function on tensile deformation has been measured[16]. Stresses in the MPa range lead to work function changes in the 10's of meV range once the elastic limit is neared or passed. The act of tuning the cathode plate may lead to QE changes due to stress induced work function changes. Part of the GTF gun tuning process involves deformation of the rear cathode plate to tune the cell. If the cathode plate is pushed past the elastic limit into plastic deformation, work function changes as much as 10's of millivolts are possible and scanning over the diameter of the plate will cause a yield variation.

Oxidation induced work function change

The work function of polycrystalline copper varies as a function of oxygen adsorption. The work function upon dosing atomically clean copper first increases, peaks and then decreases[17]. During the cleaning process, if oxygen is the exclusive contaminant, then we expect the yield (and inversely the work function) would first decrease and later increase if a wavelength near the work function is used for the yield measurement.

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