NPL Polarized Source Group
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Cathode Changing Procedures for the Illinois/CEBAF Polarized Electron Source

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In this note we document the procedures that have been developed for changing the cathodes in the Illinois/CEBAF polarized source. Note that the procedure varies slightly depending on the nature of the cathode material and its size. We present procedures for the "standard" (bulk) GaAs samples, for antimony-capped GaAs samples, and finally the *very preliminary* procedures that have been devised for the smaller $Zn(Ge_xSi_{1-x})As_2$ chalcopyrite samples. The chalcopyrite procedures will be updated as necessary after experience indicates that a fully-satisfactory technique has been developed.

Bulk GaAs Photocathodes

1. If using the sorption pump pair, attach them to the gun pump out port and cool them down for about 1 hour; if using the Alcatel, attach it to the gun pump-out port.

2. Close the straight-through valve under the gun, open the bake pump valve, then turn off the main ion pump, the bake ion pump, and the ARGA.

3. Make sure that the 1 psi pop-off valve is set properly, then let the system up to a pressure of 1 psi of dry nitrogen. It is crucial that this be from LN_2 boiloff or water-pumped nitrogen; oil-pumped nitrogen should be avoided at all costs.

4. Unbolt the stalk flange and remove the stalk. Do this very carefully to avoid twisting the bellows. Increase the nitrogen flow as required to maintain a positive 1 psi overpressure. Cover the port with clean aluminum foil.

5. Remove the old Ta cup and cathode. Label and store the old cathode.

6. We are now ready to strip the anodization layer off of the new cathode and load it in the chamber. This step and the following ones should be completed as quickly as possible so as to minimize the time the cathode is exposed to air.

   (a) Swirl the cathode, face up, in a beaker of electronic grade ammonium hydroxide for 30 seconds.

   (b) Decant off most of the ammonium, leaving just enough to cover the cathode.

   (c) Rinse 5 times with deionized water, and then 5 times with methanol, being careful not to expose the surface to air.

   (d) Remove the cathode from the methanol, blow dry it with nitrogen, and then mount it on the stalk using a fresh Ta cup, crimped over the stalk, as a clamp.

7. Place the stalk in the chamber. Make sure that the nitrogen pressure is at 1 psi and the popoff valve is set at 1 psi.

8. Seal the stalk flange, again taking care to avoid damage to the bellows. Then close the up-to-air valve, open the valve to the roughing pump (the Alcatel or the sorption pump pair), and evacuate and bake the system following the standard procedure appropriate for a bulk sample.

Antimony-capped GaAs Photocathodes

1. If using the sorption pump pair, attach them to the gun pump out port and cool them down for about 1 hour; if using the Alcatel, attach it to the gun pump-out port.

2. Close the straight-through valve under the gun, open the bake pump valve, then turn off the main ion pump, the bake ion pump, and the ARGA.

3. Make sure that the 1 psi pop-off valve is set properly, then let the system up to a pressure of 1 psi of dry nitrogen.

4. Unbolt the stalk flange and remove the stalk. Increase the nitrogen flow as necessary to maintian the 1 psi overpressure. Cover the port with a clean piece of aluminum foil.

5. Remove the old Ta cup and cathode. Label and store the old cathode.

6. Remove the capped cathode (see note) from its clean storage container, and degrease it by:

   - ultrasonic cleaning for two minutes at low power, face-up in a clean beaker of electronic grade acetone; then
   - ultrasonic cleaning for two minutes at low power, face-up in a fresh beaker of electronic-grade methanol; then
   - ultrasonic cleaning for two minutes at low power, face-up in a fresh beaker of distilled water.

7. Then blow dry the cathode with dry nitrogen, and mount it on the stalk using a fresh Ta cup, crimped over the stalk, as a clamp.

8. Place the stalk in the chamber. Make sure that the nitrogen pressure is at 1 psi and the popoff valve is set at 1 psi.

9. Seal the stalk flange, close the up-to-air valve, open the valve to the roughing pump (the Alcatel or the sorption pump pair), and evacuate and bake the system following the standard procedure appropriate for a thin (capped) sample.

Note: Before storage the capped sample should have the indium solder on the back side removed (if it wasn't done by the crystal grower). This is done by ultrasonic cleaning for about one minute in a solution made by disolving 1 gm of $HgCl_2$ in 10 ml of Dimethyl Formide, followed by rinsing in deionized water and air drying.

$Zn(Ge_xSi_{1-x})As_2$ Chalcopyrite Photocathodes

We present here the procedure used for our initial tests on chalcopyrite photocathodes. It will be updated and improved as experience leads us to a fully-satisfactory procedure.

1. If using the sorption pump pair, attach them to the gun pump out port and cool them down for about 1 hour; if using the Alcatel, attach it to the gun pump-out port.

2. Close the straight-through valve under the gun, open the bake pump valve, then turn off the main ion pump, the bake ion pump, and the ARGA.

3. Make sure that the 1 psi pop-off valve is set properly, then let the system up to a pressure of 1 psi of dry nitrogen.

4. Unbolt the stalk flange and remove the stalk. Increase the nitrogen flow if necessary.

5. Remove the old Ta cup and cathode. Label and store the old cathode.

6. If switching operation from GaAs samples to chalcopyrite samples, it is necessary at this point to disassemble the gun and replace the Pierce electrode with the revised electrode for the chalcopyrite samples (2709- 92). This will take a considerable amount of time, and will increase the duration of the bakeout cycle required after the gun is reassembled.

7. Begin the cleaning of the $Zn(GeSi)As_2$ by a three-step process of 15 minutes of ultrasonic cleaning in 1,1,1 trichloroethane, then 15 minutes in acetone, and then 15 minutes in methanol. Use the lowest power on the ultrasonic cleaner to avoid breaking the wafer. Use clean glass beakers and stainless steel tweezers for each step.

8. We are now ready to complete the cleaning of the new cathode and load it in the chamber. This step and the following ones should be completed as quickly as possible so as to minimize the time the cathode is exposed to air.

   (a) Etch the cathode for 3 minutes in a 1:1:100 solution of ammonium hydroxide, 30% hydrogen peroxide, and de-ionized water. Use a clean beaker and electronic grade chemicals.

   (b) Transfer the cathode quickly to a second beaker of pure, electronic grade ammonium hydroxide, and swirl the cathode, face up, for 30 seconds.

   (c) Decant off most of the ammonium hydroxide, leaving just enough to cover the cathode.

   (d) Rinse 5 times with deionized water, and then 5 times with methanol, being careful not to expose the surface to air.

   (e) Remove the cathode from the methanol, blow dry it with nitrogen, and then mount it on the stalk using a fresh Ta cup, crimped over the stalk, as a clamp. For the chalcopyrite samples it is necessary to install a centering "donut" on

the stalk (2709-90) first, then place the sample inside it, and then use a Ta cup with a smaller diameter (2709-89) for clamping it in place.

9. Place the stalk in the chamber. Make sure that the nitrogen pressure is at 1 psi and the popoff valve is set at 1 psi.

10. Seal the stalk flange, close the up-to-air valve, open the valve to the roughing pump (the Alcatel or the sorption pump pair), and evacuate and bake the system following the standard procedure appropriate for a bulk sample.

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VALVE LOCATION DIAGRAM