Standard Operating Procedure

(SOP)

Photoelectrode Fabrication and Area Determination

Test ID # PEC-P-1

Rev 1

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Revision History

This page documents the revisions over time to the SOP. The most recent iteration should be listed in the row space, with consecutive versions following.

Date of Revision	Page(s)/Section(s) Revised	Revision Explanation
04/05/2019	All	First Release

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3. Procedures

a. Scope and Applicability – This SOP details procedures for fabricating photoelectrodes and measuring their photoactive areas

b. Summary of Method – Photoelectrodes are fabricated for use in photoelectrochemical cells. Note that the mounting method can vary depending on the location of the electrical (Ohmic) contact (front or back) and type of substrate (glass, TCO, GaAs, stainless steel, etc.).

c. Definitions – Not applicable for this SOP.

d. Health & Safety Warning – Hydrogen gas is flammable. The work area should be thoroughly ventilated and free from any sources of ignition during these tests. Methanol is a neurotoxin that can be absorbed through the skin and contact should be avoided.

e. Cautions – Not applicable for this SOP.

f. Interferences – For the Hoffman apparatus, temperature fluctuations can introduce error, so temperature should be recorded at the time of measurement

g. Personnel Qualifications / Responsibilities – Not applicable for this SOP.

h. Equipment and Supplies –

1. Semiconductor wafer
2. Copper coil
3. Glass tube
4. Pliers
5. Diamond scribe
6. Lens paper
7. Indium-Gallium (InGa) eutectic
8. Diamond scribe (InGa contaminated)
9. Silver Paint
10. Oven
11. HYSOL 9462 epoxy
12. HYSOL E-120 HP epoxy
13. Disposable mixing vessels
14. Disposable stirrers

i. Step by Step Procedure

- Instrument or Method Calibration and Standardization –

1. Plan electrode dimensions based on cell openings, i.e. don't make the coil too big to fit through a 14/20 opening in a three-port glass cell.
2. Label electrodes to keep track of batch (run) number and location from parent wafer (e.g. Run number -#....MF846-1, MF846-2 etc.)
3. Calibrate the scanner pixel density be scanning a 1 cm square calibration standard and measuring the number of pixels contained in that square using the steps outlined in the data acquisition section. For example, this should be 892802 for a 2400 dpi scanner

- Sample Collection – Not applicable for this SOP.

- Sample Handling and Preservation –
    1. Never touch the front surface of the semiconductor, especially with bare hands.

- Sample Preparation and Analysis –
  A video demonstrating this protocol is available at the following location:https://www.dropbox.com/sh/aap7poletz9n21y/AADH3aAmJ1D-f8vROVzdusT5a?dl=0

  Sample preparation
    1. Use pliers to coil copper wire, insert in glass tubing
    2. Cleave samples using diamond scribe and lens paper
    3. For samples needing an InGa eutectic-based back ohmic contact (e.g. n-Si or n-GaAs), Use the contaminated scribe to scratch a small amount of InGa onto the back of the samples. Wipe back with a methanol soaked labwipe
    4. Mount samples on copper coil using silver paint (DON'T GET PAINT ON SEMICONDUCTOR SURFACE or edges. This may short out and/or contaminate the device.)
    5. Bake at 80° C for 20-30 minutes to drive solvent out of the silver paint
    6. Mix Hysol 9462 in a mixing vessel. An alternative epoxy is Hysol E-120 HP, which has a lower viscosity. The low viscosity can help if it is desirable to have a thin perimeter lip of epoxy to help bubbles clear from the photoelectrode. A N_2 gun may be used to help further flatten the epoxy lip after application. The low viscosity epoxy can sometimes wick further onto the photoelectrode surface to a degree that may be undesirable. This can be avoided by letting the E-120 HP sit for 45-90 minutes after mixing before using.
    7. Cover edges of semiconductor and all exposed copper wire with HYSOL 9462 epoxy
    8. Leave on benchtop for at least 3-4 hours (typically overnight). If bubbles in the epoxy are an issue (sometimes caused by too rapid

mixing of the epoxy), placing the mixed epoxy in a vacuum chamber (e.g. load lock) can accelerate removal of bubbles.
9. To ensure full epoxy cure, bake at 80° C for 2 hours
10. For additional acid resistance, an additional thin layer of HYSOL E-120 HP may be used on top of the HYSOL 9462. This epoxy is much less viscous. Allowing the mixed epoxy to sit for 45-90 minutes to increase viscosity often makes its application easier.
11. Leave on benchtop at least 3 hours
12. Bake at 80° C for 2 hours

- Troubleshooting – Not applicable for this SOP.

- Data Acquisition, Calculations & Data Reduction Requirements –

1. Place electrode on scanner face down
2. Align the electrode so the surface is parallel to the scanner glass
3. Scan the electrode at 2400 dpi resolution in grayscale
4. Process the file in ImageJ as following:
5. Image-> Adjust-> Threshold(Ctrl + Shift + T) and move the bottom threshold slider back and forth until the appropriate surface area is all red, then hit "Apply"
6. Select the area of the electrode using the magic wand tool, then hit Edit -> Fill (Ctrl + F) to fill the electrode area with black
7. Hit Edit -> Clear Outside to remove the remainder of the image
8. Open the histogram function (Analyze -> Histogram (Ctrl + H))
    a. In the histogram plot, the horizontal axis ranges from black to white which may correspond to either 0 to 250 or 250 to 0 depending on your image settings. Use this histogram to identify whether the pixel count at 0 or 250 is that of the active area that you are trying to measure.
9. Select "List" and identify the relevant pixel count in the right column, and record this number.
10. Calculate the surface area by diving the number of black pixels by the number of pixels per cm2. For 2400 dpi, the number of pixels per cm2 is (892802)

- Computer Hardware & Software – Flatbed scanner with 2400 dpi resolution at minimum. Computer for scanner control and analysis. ImageJ.

j. Data and Records Management – Not applicable for this SOP.

4. Quality Control and Quality Assurance Section

5. Reference Section