Standard Operating Procedure

(SOP)

PEC Device Fabrication/Integration and Scale Up

Test ID #PEC-P-10

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
04/01/2019	All	First Release

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3. Procedures

a. Scope and Applicability – The purpose of this SOP is to provide details on PEC cell fabrication and assembly of the cell described in: Karl A. Walczak, Gideon Segev, David M. Larson, Jeffrey W. Beeman, Frances A. Houle, Ian D. Sharp, "Hybrid Composite Coatings for Durable and Efficient Solar Hydrogen Generation under Diverse Operating Conditions", Advanced Energy Materials, 7 (2017), 16027921.

b. Summary of Method – The procedure consists of fabricating components of a PEC cell and assembling to for a PEC device.

c. Definitions –

- PEC: photoelectrochemical cell

d. Health & Safety Warning –

- Wear adequate personal protective equipment (PPE) for both the experimental apparatus as well as working outdoors, with environmental exposure.

e. Cautions – Not applicable for this SOP.

f. Interferences – Not applicable for this SOP.

g. Personnel Qualifications / Responsibilities – user should be familiar with indoor PEC testing, STH and IPCE measurements

h. Equipment and Supplies –

- Silver glue: CW2400 (Circuit works)
- Copper wire: 34 gauge
- Epoxy: Epotek 302-3M epoxy (Epoxy Technologies, Billerica, MA).
- Acrylic stock: cast acrylic 1/4"x2"x2" (McMaster-Carr)
- Titanium grid: Consisting of 0.28 mm diameter wire woven into an 18 wire per in. mesh (BeanTown Chemical)
- Photovoltaic Element: UTJ cell from Spectrolab Inc without anti reflectance coating
- Gas tubing: Upchurch PEEK tubing with an outer diameter of 1.8 mm and an inner diameter of 1.05 mm
- Gasket: Viton 75 O-rings (McMaster Carr) were used to seal each of the chambers.
- Bolts: stainless steel socket head cap head bolts (size 2–56 length 0.50 in.)
- HER and OER catalyst: Sputtered Pt and Ir respectively
- Grid assembly two point compression rig: PTFE rig

i. Step by Step Procedure

ii. Machining Cell Chassis

a. Acrylic stock

i. Cut acrylic stock sheet into appropriately sized pieces
ii. Cut gasket grove
iii. In PV carrier plate, cut opening for membrane and PV
iv. Drill holes for bolts, gas tubing, and electrode placement
v. After fabrication the components were cleaned: were cleaned in mildly agitated deionized water containing detergent for 10–20 min, after which they were rinsed in cold deionized water. A deionized water stream was used to clean out screw holes and ports. Next all parts were again rinsed in cold deionized water, rinsed with warm deionized water, and dried under flowing N2
vi. Epoxy gas ports: Cut PEEK tubing to 5mm coat the outside with epoxy and place into ports on acrylic chassis. Let dry overnight
vii. Press gasket into groove in chassis

b. Preparing working and counter electrode

i. The working electrode, hydrogen evolution reaction electrode is coated onto the Ti wire mesh that is to be placed onto the PV.
ii. Clean the Ti grid: As-received grids were solvent-cleaned by sequential sonication in acetone (BDH, semiconductor grade) and isopropanol (BDH, semiconductor grade), followed by drying under high purity N2
iii. Next use a sputtering or other coating system to apply Pt catalyst onto Ti grid
iv. For the counter electrode, a Ir mesh, Ir coated Ti grid with Ir wire current collector can be used.

c. Mounting PV cell onto chassis:

i. The PV is diced into 1x1cm piece
ii. The edge of the acrylic window is coated with epoxy
iii. Place PV on acrylic window and apply light pressure to ensure spread and contact of epoxy without covering surface of PV
iv. Let dry overnight
v. Next take cleaned, cut and coated Ti grid and place over the PV
vi. To create the composite coating, the catalyst-coated wire grid was mechanically pressed against the surface of the photovoltaic element with a custom 2-point compression unit held in place with finger-tightened nuts on threaded support rods
vii. Weighing and preparing epoxy: The epoxy was prepared by weighing the two components to a weight ratio of 100:45, following by manual mixing for 1 min. Next, the epoxy was degassed in a chamber evacuated to 150 mTorr for 5 min, after

which it was purged with nitrogen. The epoxy was then applied to the grid/photovoltaic assembly and cured in at 55 °C for 12 h in air, after which it was cooled to room temperature

d. PEC Cell Assembly
    i. Place one chassis end plate with gasket down on a clean flat surface. Next place PV carrier chassis down on top of the first endplate.
    ii. Place membrane cut to correct size over the back side of the PV chassis
    iii. Put counter electrode wire through port hole in second endplate, adjusting the height to bottom 2/3 of cell
    iv. Place second endplate over the remaining assembly aligning with the bolt holes.
    v. Place bolts through the holes, and place nuts on the back side
    vi. Carefully tighten to hand tight
    vii. Using torque wrench torque to XN.m

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- Sample Preparation and Analysis – Not applicable for this SOP.
- Troubleshooting – Not applicable for this SOP.
- Data Acquisition, Calculations & Data Reduction Requirements – Not applicable for this SOP.
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j. Data and Records Management –

Not applicable for this SOP.

4. Quality Control and Quality Assurance Section

Not applicable for this SOP.

5. Reference Section