PIKE TECHNOLOGIES

INSTALLATION AND USER GUIDE

Jackfish
Electroless Deposition Kit

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1 Introduction

The Jackfish SEC Electroless Deposition Kit is designed to allow the end-user to chemically modify the reflecting plane of a face-angled crystal (FAC) with a metal film. Chemical-resistant Teflon and Viton materials prevent corrosive solution from causing damage to the sides of the crystal.

The electroless deposition process involves stripping the oxide layer on the Si surface followed by gold deposition via the dissolution of Si as follows:

$$Si_{(s)} + 6F^-_{(aq)} \rightarrow SiF_6{}^{2-}_{(aq)} + 4e^-$$

$$AuCl_4{}^-_{(aq)} + 3e^- \rightarrow Au^0_{(s)} + 4Cl^-_{(aq)}$$

Assembled Electroless Deposition Kit.

2 The Electroless Deposition Kit

In order for you to quickly verify receipt of your accessory, we have included a packing list. Please inspect the package carefully. Contact PIKE Technologies immediately if any discrepancies are found.

2.1 Packing List

Electroless deposition PTFE mask

P.N. 162-4733
Qty. 1

60 degree FAC holder, PEEK

P.N. 162-4745
Qty. 1

#8-32 screw, stainless steel, 1-1/2" long

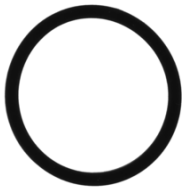
Qty. 8

#8-32 nut, stainless steel

Qty. 8

-018 square profile FKM o-ring

Qty. 2



9/64" hex L-key

Qty. 1

3 Instructions

3.1 Deposition Solution Preparation

CAUTION! The following solutions are extremely corrosive and present a significant health hazard. Use proper PPE and follow all relevant safety protocols when working with these chemicals.

The procedure described here was first described by Osawa in [1] and further modified in [2]. Recent use of this recipe on a FAC is described in [3].

Make all solutions with ultrapure water and analytical grade chemicals.

- 40% wt NH_4F – used to remove the oxide layer from the Si reflecting plane. Remake this solution regularly.
- Buffer Solution (0.3 M Na_2SO_4, 0.1 $Na_2S_2O_3$, and 0.1 M NH_4Cl) – can be stored for several months of use.
- 2% HF – reduces the gold salt during the deposition process. Can be stored (in an appropriately compatible container) for several months of use.
- 0.015 - 0.030 M $HAuCl_4 \cdot 3H_2O$ – gold solution to be prepared fresh before deposition. Other gold salts may also be used.

3.2 Crystal Preparation

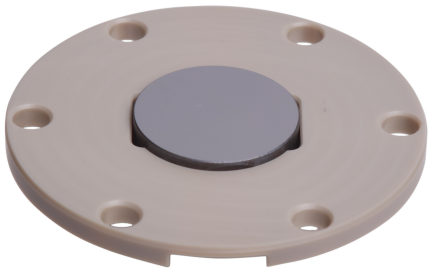
Polishing the crystal is extremely important for high quality gold films. During the following steps, dust or debris on the crystal surface should be strictly avoided.

1. Using successively finer alumina slurries, polish the reflecting plane of the FAC for 20 minutes on each polish grade, rinsing with copious amounts of ultrapure water in between. *Polishing should be completed immediately prior to gold deposition!* We recommend two steps – one with 3 µm polish and another with 0.5 µm polish. Osawa and coworkers have reported using 0.03 µm polish (see [1]).
2. Sonicate the FAC in solvent (ethanol or acetone) and then in ultrapure water. Ensure that the FAC faces are carefully covered/protected during sonication to prevent damage to the crystal from contacting the walls of the sonication vessel.
3. Rinse with copious amounts of ultrapure water to remove all traces of solvent or debris. *Do not touch the reflecting plane during rinsing.*

3.3 Assembly of the Electroless Deposition Unit

Place the O-ring in the groove in the Teflon deposition mask.

Place the face-angled crystal (FAC) in the FAC holder and make sure the polished reflecting plane protrudes slightly above the top plane of the holder. Take care not to touch the reflecting plane.



Place the Teflon deposition mask, O-ring side down, onto the FAC. Ensure that the O-ring sits on top of the polished reflecting plane along the entire circumference of the FAC. Align the clearance holes on the FAC holder and deposition mask and place the screws in each of the six holes.

After placing a nut on each screw, hand tighten each of the six nuts. Then tighten with a screwdriver in a star pattern to ensure the deposition mask is even with the FAC holder. It is important to ensure that the mask does not shift from the FAC holder during this process.

Note: Once the assembly is complete, leak check the fixture by filling the well formed by the Teflon deposition mask and the surface of the FAC with water. Allow this to sit for at least 30 minutes and ensure there are no leaks prior to proceeding with corrosive solutions. This is especially important when using the Electroless Deposition Kit for the first time.

The O-ring size was chosen to maximize the exposed area of the FAC reflecting plane. Any error in assembly can cause solution to leak onto the sides of the crystal which will have detrimental affects on surface quality.

When the assembly is complete, the long end of the screws should be coming out of the bottom of the FAC holder, as shown in the figure above. These will serve as a stand for the fixture.

Prepare a water bath held at 55°C with stirring. Ensure that the water level in the bath is sufficient to completely cover the bottom half of the FAC from the underside of the fixture but low enough that it does not spill onto the FAC reflecting plane. Allow the FAC to come to the proper temperature before continuing.

3.4 Deposition

1. Freshly prepare the gold salt solution described in Section 3.1. We recommend about 1 mL of solution.
2. With a glass pipette, cover the FAC reflecting plane with NH_4F solution. Allow this solution to react for 3 minutes.
3. While waiting, add to a Teflon beaker (or other non-reactive beaker):
   (a) Roughly 2 mL of the buffer solution
   (b) 1.1 – 1.3 mL 2% HF solution. We recommend using a plastic syringe to measure this volume and mix it into the buffer solution.
4. When the three minutes have elapsed, add the gold salt solution to the beaker and mix well (this is the *plating solution*).
5. Empty the NH_4F from the fixture.
6. Gently place 1.0 mL of plating solution on the FAC reflecting plane, avoiding bubbles.
7. Wait 8 to 10 seconds and gently remove 0.5 mL of plating solution from the FAC reflecting plane. Add another 0.5 mL.
8. Repeat Step 7 until all of the plating solution has been used.
9. Quench the reaction by removing the Electroless Deposition Kit from the water bath and rinsing with at least 0.5 L of ultrapure water.
10. Disassemble the Electroless Deposition Kit and rinse the gold-modified reflecting plane for at least 30 seconds with ultrapure water.
11. Carefully dispose of all waste and clean all components which have been exposed to corrosive solutions. We recommend prolonged immersion in ultrapure water in addition to any institutional protocols.

3.5 Fine-tuning the Process

This process requires significant practice and skill to achieve high-quality films. When troubleshooting, consider the cleanliness of the crystal and all chemicals and labware used in the process. If the film appears thick or uneven, then shorter deposition intervals in Step 7 of Section 3.4 can be tried, ranging from 4 to 6 seconds. Alternatively, the gold concentration can be lowered. If the film is still uneven, try a longer polishing procedure or with finer grade polish.

Gold films deposited in this fashion are extremely fragile – great care must be taken when assembling the Jackfish SEC J1 spectroelectrochemical cell to avoid damaging the gold layer.

4 Support

This manual and future updates are made available online at https://jackfishsec.com/support.

We have a blog which addresses some common questions such as preparing internal reflection elements for ATR-SEIRAS and selecting the optimal angle of incidence. Check it out at https://jackfishsec.com/blog.

We'd love to hear from you! Questions and feedback can be directed towards info@jackfishsec.com.

References

(1) Miyake, H.; Ye, S.; Osawa, M. Electroless Deposition of Gold Thin Films on Silicon for Surface-Enhanced Infrared Spectroelectrochemistry. *Electrochemistry Communications* **2002**, *4* (12), 973–977, DOI: 10.1016/S1388-2481(02)00510-6.

(2) Rosendahl, S. M.; Danger, B. R.; Vivek, J. P.; Burgess, I. J. Surface Enhanced Infrared Absorption Spectroscopy Studies of DMAP Adsorption on Gold Surfaces. *Langmuir* **2009**, *25* (4), 2241–2247, DOI: 10.1021/la803404u.

(3) Sigrist, J.; Lins, E.; Morhart, T.; Briggs, J.; Burgess, I. Optimization of a Commercial Variable Angle Accessory for Entry Level Users of Electrochemical Attenuated Total Reflection Surface Enhanced Infrared Absorption Spectroscopy (ATR-SEIRAS). *Applied Spectroscopy* **2019**, *73* (12), 1394–1402, DOI: 10.1177/0003702819858353.

5 Also Available From Jackfish SEC

J1 Spectroelectrochemical Cell

Our flagship cell. Features a separate reference arm and glass construction for ultimate cleanliness.

J2 Spectroelectrochemical Cell

A completely sealed cell with a simple design. Compatible with both aqueous and volatile solvents.

J3 Easy Electrochemistry "EEL" Flow Cell

A cell with a small internal volume and flow capabilities, allowing researchers to investigate complex and dynamic surface phenomena.

J4 H-Cell

A cell with two glass compartments separated by a user-supplied membrane. Includes a quartz window for visual inspection of the working electrode.

Si Microgrooved Wafers

Uncoated microgrooved silicon wafers, available in a choice of two different face angles: 35° and 55°.

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