Small Devices for Photo-Induced Electrochemical Synthesis

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Primary CNF Tools Used: Heidelberg mask writer - DWL2000, ABM contact aligner, Oxford 81/82/100/Cobra etchers, AJA sputter deposition tool, Oxford PECVD, hot press, Unaxis 770 deep Si etcher

Abstract:

We present microscopic (hundred-micron scale) wireless electrochemical synthesizers that allow for high throughput experimentation (HTE) of small (<100 uL) volumes, allowing for more rapid development of electrochemical synthesis methods in the pharmaceutical industry. Current methods of electrochemical HTE for drug synthesis require epoxying macroscopic electrodes to a custom printed circuit board, placing a lower limit on the reaction volume and limiting the ability to scale to 96- or 384- well plates. In contrast, these Small devices for Photo-induced ElectroChemical Synthesis (SPECS) are individually manipulable and optically powered, allowing for more efficient parallelization of the drug synthesis process.

Summary of Research:

SPECS allow for completely wireless electrochemical synthesis. These devices are fabricated using standard photolithographic techniques, and thus can be produced at the wafer scale. The only serial process in SPECS fabrication is their individual manipulation after separation from the substrate, which is easily automated with a commercial pick-and-place tool.

This technology uses microscale silicon photodiodes connected in series with two electrodes to convert incident light to a current and voltage that can be used to induce electrochemical synthesis at the electrodes. The entire device is encapsulated in 1 uL of silicon dioxide except for the electrode surface which remains exposed to the environment. Under an illumination intensity of about 100 nW/μm^2 at 532 nm, a 100 μm diameter device consisting of seven photodiodes can produce about 20 μA and and 4.5V. The current produced by the SPECS increases linearly with incident light intensity and device area, with the voltage increasing linearly with the number of photodiodes in series — this allows the device to be tailored to the desired synthesis process while still allowing for *in situ* tuning.

To create the silicon photodiode devices, we begin by selectively doping the top of the SOI device layer with phosphosilicate glass to create a vertical PN junction. We then electrically isolate the photodiodes by dry etching to the buried oxide layer in the Oxford Cobra inductively coupled plasma (ICP) etcher. We then connect the photodiodes in series to each other and the electrodes, also conformally coating the silicon defining the electrodes in metal at this step. The metal electrodes and interconnects are platinum with a titanium adhesion layer deposited in the AJA sputter deposition tool.

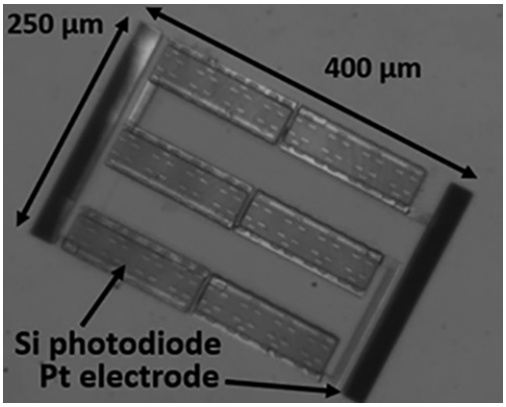

Figure 1: A prototype SPECS device, with six photovoltaic panels and one platinum electrode on each end.

We encapsulate the photodiodes with silicon dioxide with the Oxford plasma enhanced chemical vapor deposition tool, leaving the metal electrodes protruding (Figure 1).

We then sputter and pattern aluminum over the entire device except for the center of the photodiodes. This prevents light from passing through the transparent sections of the device during experiments, which could result in unintended photo-induced synthesis of the reaction mixture.

Due to using planar lithographic techniques to create the SPECS, they are 5 μm thick as fabricated. These thin devices can be released from the substrate by patterning aluminum over them, undercutting the silicon underneath with XeF_2, and then releasing in wet aluminum etchant. After releasing in fluid, the devices are easily pipetted into any container for experimentation (Figure 2). Preliminary results have been achieved with these devices (Figure 3).

To increase rigidity of larger (mm-scale) devices, we transfer them to a transparent substrate. To accomplish this, we bond the wafer with the onboard SPECS to a transparent carrier wafer using a low melting-point thermal plastic polymer and then remove the silicon handle substate via ICP dry etching in the Unaxis 770 Si deep etcher. In the end, we attach the carrier wafer/SPECS stack to a glass substrate and remove the carrier substrate by melting the bonding polymer, followed by dicing to separate the individual devices.

Future work includes electrochemical synthesis using these larger and more rigid SPECS, as well as characterization of their rigidity during sonication, stirring, and other methods of regulating mass transport *in situ*.

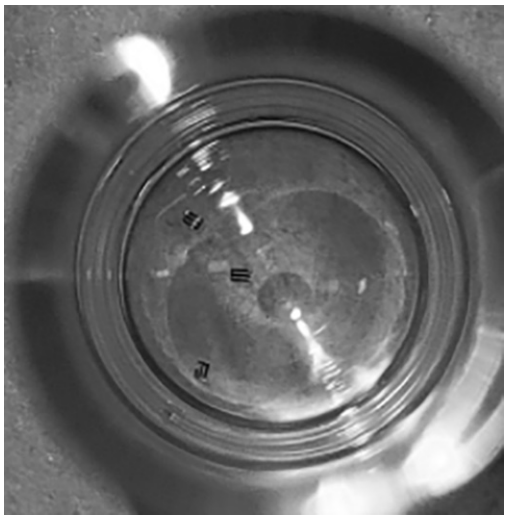

Figure 2: Multiple SPECS after being pipetted into a 5 mm diameter glass vial to be inserted into a 96 well plate.

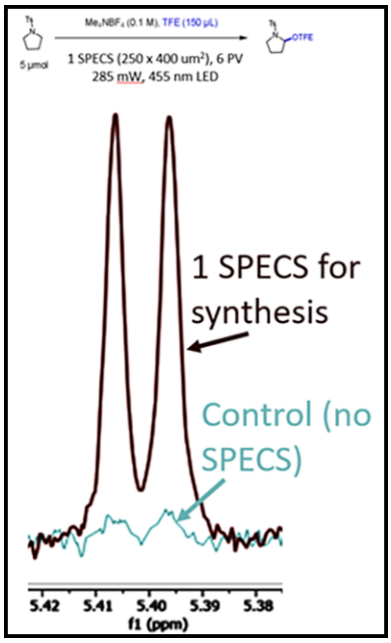

Figure 3: Nuclear magnetic resonance spectroscopy results showing successful Shono oxidation using SPECS, a standard electrochemical synthesis reaction.