Electronic Supplementary Material (ESI) for Energy & Environmental Science.

Supplementary Materials for

Investigation of thin/well-tunable liquid/gas diffusion layers exhibiting superior multifunctional performance in low-temperature electrolytic water splitting

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Nano-manufacturing of titanium thin/well-tunable LGDLs

A typical fabrication procedure for thin titanium LGDL begins with the design and fabrication of the photomasks, (as shown in Fig. S1), which is the most important step to control the pore size, pore shape and porosity of LGDLs. A mask pattern was designed using commercially available CAD/VLSI software (LayoutEditor, layouteditor.net). The design pattern was imported into a Heidelberg DWL 66 laser lithography system and patterned on a soda-lime glass mask plate that is pre-coated with chromium and photoresist. After patterning, the masks were developed for 1 minute in Microposit® MF® CD-26 Developer (Shipley Company, Marlborough, MA), rinsed with DI water and dried in N_2. Masks were then submerged in chrome etchant for 2 minutes, rinsed with DI water and dried in N_2. The remaining resist was subsequently removed in a heated bath (70 ^{o}C) of N-methyl pyrolidone (NMP). The titanium thin film was placed on the resist-coated silicon wafer with special care due to its delicate features, and gently heated for 90 s at 115 ^{o}C. A second layer of adhesion promoter (MicroPrime MP-P20, ShinEtsuMicroSi) and MEGAPOSIT™ SPR™ 220 photoresist (MicroChem) was applied to the titanium foil under identical conditions, and then exposed to UV light using conventional contact photolithography. It was then developed in Microposit® MF® CD-26 developer (Shipley Company, Marlborough, MA), rinsed with DI water and dried in N_2. After patterning the photoresist mask on the foil, the patterned material was etched in HF. The photoresist was the removed, completing the processing of the thin titanium LGDL.

Introduction of the high-speed and micro-scale visualization system

To observe the phenomena of electrochemical reaction inside an operation PEMEC, a high-speed and micro-scale visualization system (HMVS) was built and used with a transparent PEMEC by taking advantage of new LGDL development, which is shown in Fig. S2. To get the visual image from inside of PEMEC, the following modifications were made to the conventional electrolyzer cells: (i) a rectangular hole was cut into the anode end plate as an observation window; (ii) the copper anode current distributor was removed; and (iii) the graphite anode bipolar plate with a parallel flow field was separated into two parts, one was a transparent block with flow-in hole, the other one was a thin titanium plate serving as the flow channel. In the transparent electrolyzers, the anode LGDL flow fields with current distributors were made chemically through etching titanium plates to form flow channels with lands for current distributions. They were capped by transparent plates and visually accessed through a rectangular window in the aluminum end plate. A 25 μm titanium thin film with 791 μm circular pores was installed as the LGDL during operation of PEM water electrolyzer. The channel width was 1061 μm. These changes allow for optical imaging of the anode. A high-speed micro-scale visualization system was possible using a high speed camera Phantom V711 and long-distance optical system. With the V711, a maximum speed of 7530 frames-per-second at full resolution can be achieved. At reduced resolutions, the camera can deliver up to 680,000 frames-per-second or up to 1,400,000 fps with the FAST option. With all this equipment and design, local O_2 formation can be monitored and analyzed based on micro-scale bubble formation in transparent/operational PEMECs by HMVS.

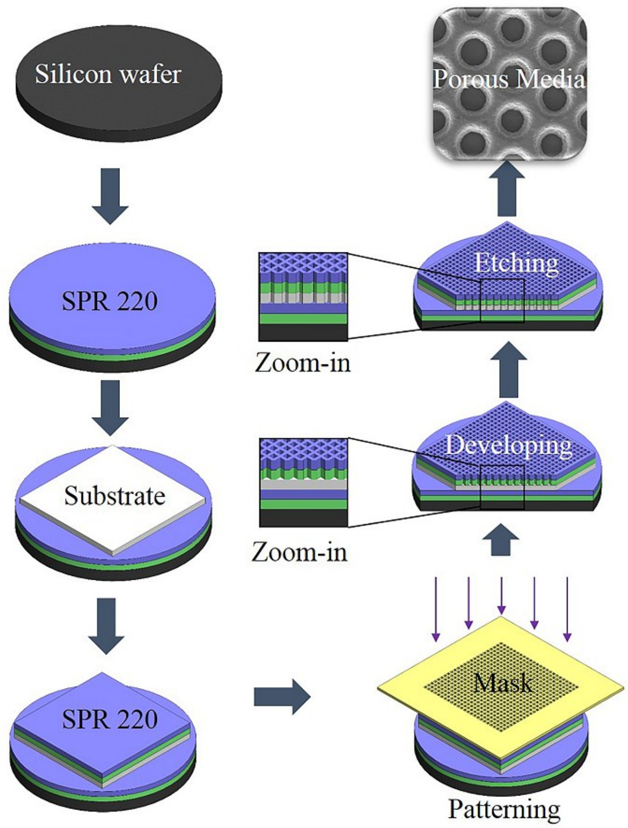

Fig. S1 Typical fabrication process for thin titanium LGDLs

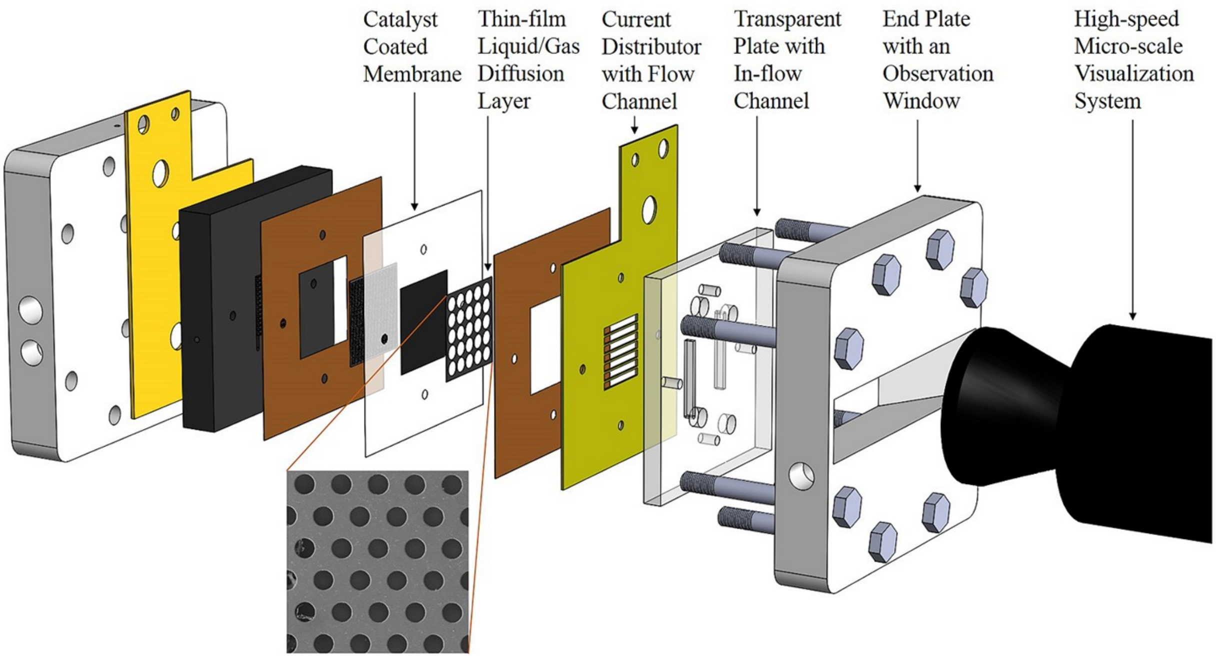

Fig. S2 Schematic of high-speed micro-scale visualization system (HMVS) and transparent PEMEC with thin film/well tunable LGDL