University of California at Berkeley
College of Engineering
Mechanical Engineering Department

ME138/238, Spring 2018 **Liwei Lin**

Microfluidics & Water Splitting Lab
Assigned: February 20th; Due: March 15th

Contacts: Eric Sweet (ericsweet@berkeley.edu) and Emmeline Kao (kao@berkeley.edu)
If you have any questions, feel free to contact us!

Engineering a Microfluidic Water Splitting/Hydrogen Production Device

Welcome to the final lab of the course! Over the next few weeks, you will be designing, fabricating and characterizing your own prototype microfluidic device that could, in theory, be used to perform water splitting and hydrogen production/collection in a single device. You will need to design the microfluidic structure, perform PDMS casting using a 3D printed mold, assemble the components, and conduct experiments to validate if hydrogen production was successful or not. After the completion of the design and fabrication process (covered in lab session #1), you will need to fill your device with water, expose to an excitation light source, harvest the produced gases, and test for the presence of hydrogen.

Your goal will be to use generate hydrogen through the water splitting reaction, route the hydrogen gas via your PDMS mold, and gather the hydrogen gas. Please read through Paper #1 carefully as the PDMS casting protocol is based on the same process; likewise, please read through Papers #2a,b which describe the theory behind water splitting and a similar method of microfluidic hydrogen generation, respectively.

IMPORTANT: This lab requires planning and organization, not unlike a real MEMS experiment. We have outlined a schedule for you with multiple intermediate deadlines. Be sure to plan ahead, so that you don't miss any intermediate deadlines. If you do, it will be *extremely* difficult to complete the entire lab. Additionally, attendance at these lab sections will be part of your grade. You will lose 10% on your individual lab report grade per section missed (see Section C for grading details). Here's the general schedule,

(1) **Week of 2/19-2/23**: Brainstorm, sketch and design a 3D print-able mold to cast the PDMS microchannel structure. Using either Solidworks or AutoCAD, create a 2D profile (sketch) to define the microchannel layout, then extrude in 3D to create a 3D model of the physical mold. **Hint**: carefully follow the design guidelines provided in lecture to create a physical 3D mold suitable for PDMS casting (with your group's name somewhere on the part.

(2) **Thursday, 2/22 (5-6:30pm)**: Go to 1113 Etcheverry and the GSI's will provide a demonstration of the *PDMS casting method using a 3D printed mold*. Take detailed notes on the protocol, procedure. You should ask the GSI any questions regarding PDMS dimensions or thickness, curing time and temperature, mold preparation, how to store the PDMS part, etc.

(3) **Friday, 2/23 (by 5pm)**: Email the GSI (ericsweet@berkeley.edu) your CAD model. He will print, collect and deliver the models to 1113 Etcheverry on 2/26.

(4) **Thursday, 3/1 (5-6:30pm)**: Bring your **completed** PDMS structures, go to 125 Stanley Hall and the GSI's will *bond your PDMS structures to your silicon*

wafers, as well as provide a demonstration of how to *fill your devices with water, excite the wafer, collect the resulting gases, and measure the results*.

(5) **Week of 3/5-3/9**: On your normally assigned lab sections (see Prof. Lin's website for the section schedule), learn water splitting measurements from GSI in 1113 Etcheverry. You will be operating the Gamry, see Section A for detailed protocol and help troubleshooting.

(6) **Tuesday, 3/13 (6-6:30pm)**: Bring your vial of produced gases to 1113 Etcheverry Hall. The GSI will determine the presence of H2 *qualitatively* :)

(7) Due **Thursday, 3/15 (668 Sutardja Dai Hall; 5:10 pm)**: Submit your lab report, following the template given in Section C, in the mailbox outside of 668 Sutardja Dai Hall by 5:10 PM. All lab reports after this time will be considered late and penalized accordingly (see Section C). Your lab report should include design, fabrication steps and results, experimental procedure and results, discussions and other observations. Specifically discuss what (fabrication error? quality of photoelectric substrate? protocol or measurement error?) are the primary reasons for the lack of observed hydrogen production **and potential design/experimental protocol changes** that could improve the performance.

(8) Due **Thursday, 3/15 (email to kao@berkeley.edu; 11:59 PM):** If you are working in a group of 2 or more you must complete a team evaluation (see Section C for details). This is mandatory for all teams: you will lose 15% of your individual lab grade if you do not turn it in. Email this to kao@berkeley.edu by 11:59 PM.

For Graduate Students:

(9) Conduct analytical and/or numerical simulations to study the design optimization of your microfluidic channel(s), including optimized dimensions with possible validations from experimental results.

(10) Discuss potential applications for this device, and corresponding design improvements that would need to be made for the prototype to best meet the needs of the specific applications (packaging? device interfacing hardware?)

(11) Discuss and provide specific reasonings/explanations/design concepts: instead of a serving as a simple hydrogen production tool, can your device be used to generate electrical current (hydrogen fuel cell?)

	Monday	Tuesday	Wednesday	Thursday	Friday
W1	2/20	2/21 *Lecture*: Microfluidics (Eric)	2/22	2/23 *Lecture*: Water Splitting (Emmeline)	2/24 **CAD Model Due**
W2	2/26	2/27 *Lab (1113 Etch.)*: PDMS casting (5-6:30pm)	2/28	3/1 *Lab (125 Stanley)*: Wafer bonding (5-6:30pm)	3/2
W3	3/5	3/6 *Gamry Experiments* ("Lab time")	3/7 *Gamry Experiments* ("Lab time")	⅜ *Gamry Experiments* ("Lab time")	3/9
W4	3/12	3/13 *Demonstration* (1113 Etch.)	3/14	3/15 **Final Report Due**	3/16

Summary of deadlines and mandatory meetings:

- 2/23 5–6:30 PM - entire class learn PDMS casting in 1113 Etch
- **2/23 11:59 PM - final CAD outline of PDMS mold due to ericsweet@berkeley.edu**
- 2/28 5 PM - have your PDMS mold casted. It needs at least 24 hours to cure before we plasma bond
- 3/1 5–6:30 PM - entire class meet in 126 Stanley for plasma bonding
- 3/13 5 PM - have the vials of (maybe) hydrogen ready to test for hydrogen presence!
- **3/15 5:10 PM - final reports due to mailbox outside 668 Sutardja Dai Hall**
- **3/15 11:39 PM - evaluations due (PDF or plain text email ONLY) to kao@berkeley.edu**

Section A: Gamry Operation

The Gamry is a potentiostat, capable of measuring and manipulating voltage between 2 electrodes (working and counter), using a reference electrode. For this experiment, the working electrode will be your device. The counter electrode will be platinum. The reference electrode is Ag/AgCl (why do we need a reference electrode?)

1. Make sure the Gamry leads are all there and that black is connected to the Faraday box. The Gamry has 5 leads, all labeled in different colors: blue, green, red, white, yellow, black. You can find a mousepad with a legend to the different colors. Working and counter *actuate* the voltage, relative to the reference electrode. Working sense and counter sense *read out* the voltage between the two. Ground will be connected the Faraday box at all times.
2. Next, connect the light source. Connect the transformer to the outlet (transforms out 110 V to 220 V, since the power source is from Japan). Connect the power source to the transformer (be sure to connect it to 220 V and that the switch is set correctly to transform 110 V to 220 V). Lastly, connect the light source to the power source. Flip the red switch on the power source to warm up the source. **DO NOT hit the light button yet**, as this will turn on the light.

3. Connect working and working sense to your device, counter and counter sense to the platinum wire, and reference to the Ag/AgCl by simply clipping onto the wire/substrate/etc. You may need to use extra wires and clips as extension wires. Use tape to fix the electrodes to the beaker, if necessary.
4. Insert the platinum wire into your device, ensuring that it is not touching the substrate (or else it might short).
5. Hook up the syringe pump with water as you learned in Thursday's (3/1) lab.
6. Cover the entire setup with the black box in order to isolate it from ambient light.

Now you are ready to begin measurements! Log into the computer on the table to the left of the Gamry. The password, if you need it, is LinAdmin99.

7. Open Gamry Electrochem from the desktop. It should register the Gamry on the small bar below the taskbar.
8. Run a linear sweep scan from 0–1 V, the scan rate should be 50 mV/s. Per what you learned from the GSI in your training session, you can play around with other parameters, but do not change parameters such that Gamry would be in danger of shorting.
9. Decide whether you are taking a dark scan or a light scan and name your file appropriately. Check again that the black box is covering the entire set up. If you are running a light scan, turn on the light source at this time.
10. Run your scan! Watch it go!
11. Run an open circuit to allow it to return to its equilibrium condition. If you don't, you might see effects from the previous scan on the next scan. Run open circuit until it stabilizes (stays within 10 mV).
12. Repeat until you've finished your scans. I recommend always doing a dark after light to rule out the possibility of degradation giving you weird results.

You can do most of your analysis and data collection in EChem Analyst.

13. Open EChem Analyst from the desktop. Open your files. You can open them in overlay mode (plots multiple scans on the same plot) or one at a time.
14. In overlay mode, you can choose to see or unsee certain plots, to help your analysis.
15. To copy your data as a CSV, go to the icon on the top left, it will copy all the data on your plot to a clipboard (i.e.: if you overlay, you will get all that data).
16. Either paste the data into an excel spreadsheet or a google sheet and copy it onto a USB or email it.

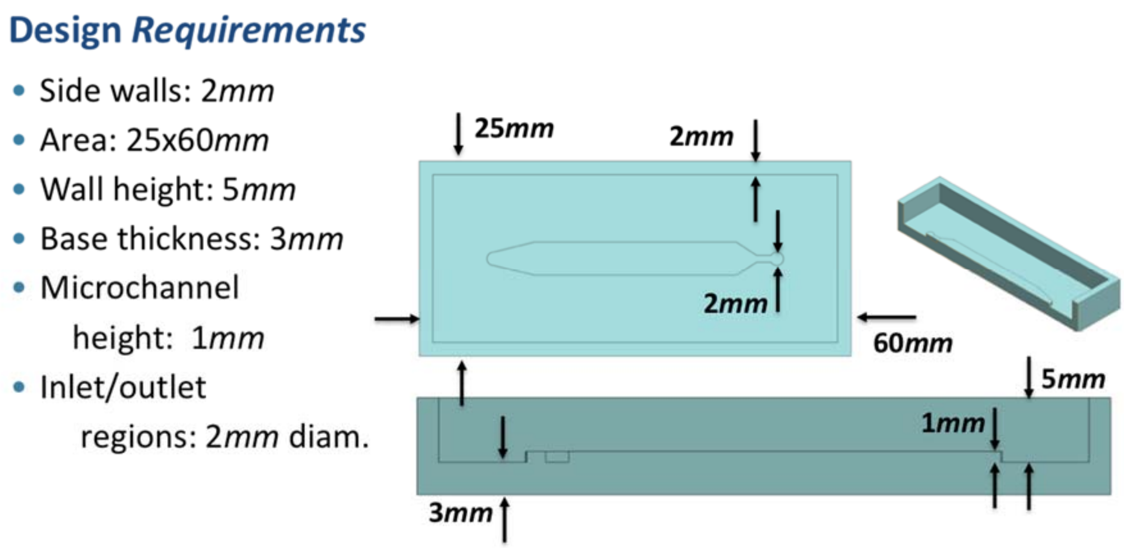
Troubleshooting:

- If you get a bunch of red dots, like the Gamry is reading past the max current, either your max I setting is too low or your leads are touching each other. Check the electrodes, wiring, etc, to make sure nothing is touching before playing around with the max I setting. You want to be especially careful of the setting so you don't short out the Gamry.
- If you get a reading remarkably close to 0 (the scale is nano or pico Amperes), recheck your wiring. Something is impeding your signal.

- If you get a reading that looks like you have double or triple samples, it is likely because the Gamry is sampling so fast that it can't catch what's going on so it's making up readings every other point (or every third point, etc.). Lower your sampling rate in this case.
- If all else fails, turn it on and off again!

Section B: 3D printed mold CAD design requirements

Please follow these specifications exactly!



Section C: Grading details

For the lab report, follow the template emailed to you (in .doc format) on 2/20 by Eric, copied below. The lab report will be graded according to the rubric, copied below.

Lab reports will be given one grade, shared between the team members. However, grades may be adjusted on an individual level based on these factors:

> **Team Evaluation:** Every team member must write a written evaluation (PDF or plaintext email ONLY), emailed to kao@berkeley.edu by 3/15, 11:59 PM. You must evaluate both (a) yourself (100–200 words) and (b) your teammate (100–200 words; 200–400 if you are working in a group of 3) on
>
> - How much each team member contributed to the team
> - The extent to which each team member's attitude, preparation, leadership, etc. contributed to the work
>
> All team member evaluations will be kept confidential. This will be ungraded, but may be used to adjust grades.
>
> **Late policy:** For all late items, you will be docked 10% of your grade for every late day. After 4 days, you will receive a 0.

Attendance: Since there are multiple components to this lab, in order to ensure that each lab member is learning the required material, we will take attendance at each lab section (2/27 5–6:30 PM, 3/1 5–6:30 PM, and during your assigned times 3/5–3/9 according to Prof. Lin's website). For every session missed, you will lose 10% of your individual lab report grade.

Section D: PDMS Bonding Results (Tuesday 3/5)

- RIE (Reactive Ion Etching) O2 plasma bonding machine protocol used:
    - 35% power (~200W); 25% O2 flow rate; 10 minutes exposure time
- Removed both Si wafer, PDMS from chamber; applied pressure for ~60 seconds by hand to allow covalent bonds to form
- Placed on hot plate (~100C) for 5-10mins

- Results:
    - Poor bonding achieved for all group's PDMS parts
    - PDMS lifted off Si wafer with little/no force

- Discussion/comments from the GSIs:
    - PDMS-SiO2 bond is known to be very hard to achieve, even using long exposure to O2 plasma
    - **Research WHY PDMS and SiO2 do not bond well. Discuss the reasons why in the discussion section of your report**
    - GSI's procured Si wafers with a native SiO2 layer on the upper surface
    - **Research potential solutions/work-arounds to achieve assembly of a successfully leak-free PDMS/SiO2-coated Si wafer microfluidic device. Discuss in the discussion section of your report**

TEMPLATE MANUSCRIPT PREPARATION GUIDELINES FOR TRANSDUCERS 2015

J.Q. Public[1], J. Doe[1], and G.I. Joe[2]
[1]The Technical Manuscript Design Group, Stanford, CA, USA
[2]The Scientific Paper Formatting Initiative, Boulder, CO, USA

ABSTRACT

The manuscript should start with a brief abstract of approximately 100 words summarizing the main goals, developments, and achievements of the work. Consider that the abstract may be included in abstract search databases. Think of what requirements the abstract should fulfill in view of this perspective.

KEYWORDS

A few meaningful keywords describing the essential topics of the paper.

INTRODUCTION

Manuscript Length

Your manuscript is expected to have a total length of four (4) pages. Longer manuscripts will be printed only if accompanied by a mandatory per page over-length charge of $250 per page. The absolute page length with payment of the over-length charge is six (6) pages. *Note that this sample is a representative of the expected manuscript length.*

The manuscript that you prepare will be printed as it is received. The electronic digest will be produced in color. There will not be a hardcopy book.

General Layout

The Technical Digest will be published in 8.5" x 11" format. Define .75" wide left, right, and top margins. The bottom margin must be 1". All paragraphs are to be indented 0.25". *Please check that the size converted correctly for your computer. Some computers do not convert the page size automatically.*

Define a two-column layout, with a space of 0.25" between columns. The title/author/affiliation section should be centered above both columns. **NO blank** line between authors and institutions.

If you choose to use another program other than Word to develop your manuscript/source file, the format MUST match the Sample/Template or it will NOT be accepted.

Since IEEE is the technical sponsor of this meeting, you are required to submit your paper through the IEEE eXpress process to create your PDF. **You should double check that your final PDF paper size is correct before uploading it to IEEE eXpress.** *Hint:* You may need to change your printer default settings. Just printing the PDF from Microsoft Word may not work. To check your page size, in Acrobat under Preferences >Page Display >Page Content and Information; click the box labeled "Always show document page size". This will display the PDF page size for your document so you may confirm the page size is correct.

Text Formatting

The Technical Digest will be printed from the PDF file which you will submit to the web-site. Your paper formatting and style MUST match the sample exactly to ensure inclusion in the Digest.

Please use Times New Roman throughout the entire manuscript, from title, authors, affiliation, headers, and sub-header, to figure and table captions, and references. To achieve a unified look across the Digest, the following formats should be used for the main paragraph types, as illustrated also by this sample manuscript:

• TITLE:	**12 POINTS, BOLD, ALL CAPITALS;**
• Authors:	*12 points, italic;*
• Affiliation:	12 points, regular;
• ABSTRACT HEADINGS:	**12 POINTS, BOLD, ALL CAPITALS, WITHOUT NUMBERING;**
• SECTION HEADINGS:	**12 POINTS, BOLD, ALL CAPITALS, DO NOT NUMBER;**
• Sub-section headings:	**11 points, bold, without numbering;**
• Text body:	10 points, regular; all paragraphs indented 0.635 cm (.25");
• Figure captions:	*10 points, italic;*
• Table captions:	*10 points, italic;*
• References:	10 points, regular, numbered.

All manuscripts must be typed single spaced to fit within the two column borders. Do not use smaller print than 10 point. Do not add any kind of pagination anywhere in the paper. Do not add a blank line between paragraphs in a section. Do not use footnotes. If you have no other option and must us a footnote, number the footnotes separately in superscripts[1]. Place the actual footnote at the bottom of the column in which it is cited. Do not put footnotes in the reference list or exceed the margins limits.

After creating your manuscript you must convert it to a PDF. You are responsible for reviewing your PDF to ensure it looks as it did in the source file you created. **Pay close attention to special characters, as these may not convert correctly.** It is important that all fonts are embedded.

ABBREVIATIONS AND ACRONYMS

Define abbreviations and acronyms the first time they are used in the text. Abbreviations such as IEEE, SI, MKS, CGS, sc, dc, and rms do not have to be defined. Do not use abbreviations in the title or section headers unless they are unavoidable.

FIGURE AND TABLE FORMATTING

All figures should be placed as close to their mention as possible. Digital images, e.g., schematic drawings, photos, micrographs, etc., should have a resolution of at least 300 dpi. Each figure should be accompanied by a numbered caption, as shown in Fig. 1, placed right below the figure being described. Figure 2 shows a photograph with unfavorable resolution (72 dpi) and low contrast.

Figure 1: Reflections on corrugated liquid/gas interface, with obstacles. Rendering with 300 dpi.

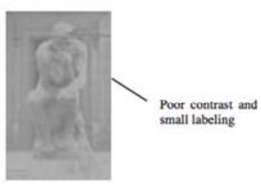


Figure 2: Reflection on life, originally shaped by Rodin: rendering with low resolution (75 dpi), unfavorable size, and other deficiencies.

Tables should span one column and should be preceded by a caption. Table 1 illustrates a possible design. Design details are left to the authors. If a table or figure is too wide to be contained in a single column, extend it over both columns, preferable at the top or bottom of a page.

Table 1: Number N of abstracts submitted and number M of submitting countries.

Region	**N**	**M**
Americas	305	5
Asia/Oceania	555	11
Europe/Africa	309	25
Total	**1169**	**41**

EQUATION FORMATTING

When numbering equations, enclose numbers in parentheses and place flush with right-hand margin of the column, i.e., with appropriate punctuation.

$$-1 \approx e^{i22\ 7} \qquad (1)$$

The above equation is meant as an example only and has no reference or further content. **Please double check that your figures, tables, etc. are numbered in correct sequence.**

REFERENCE FORMATTING

Refer to the reference section of this sample paper, for samples for contributions to Conference Digest [1], journals [2], and books [3].

If you have any questions regarding your final manuscript, please do not hesitate to contact us at sgalloway@pmmiconferences.com.

ACKNOWLEDGEMENTS

Properly acknowledge funding agencies. Also, acknowledge anyone that has assisted with your research and is not listed as a co-author.

REFERENCES

[1] A.B. Author, C.D. Author, and E.F. Author, "Title of Article, Enclosed in Quotation Marks", Thin Solid Films, 206, 94 (1992).

[2] A.B. Author, C.D. Author, and E.F. Author, "Title of Article, Enclosed in Quotation Marks", Technical Digest of the 1994 Solid-State Sensor and Actuator Workshop, Hilton Head Isl., SC, 6/13-16/94, Transducer Research Foundation, Cleveland (1994), pp. xx - xy.

[3] M. Crichton, *Prey*, Harper Collins Publishers, Inc., New York, 2002.

CONTACT

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Final Report Rubric

Grade /Maximum Points:	Characteristics:
/10	**Background: fully cited, no claims without proper citation. Is the motivation convincing?**
/10	**Research questions/claims are relevant, non-trivial, and reasonable given background information**
/5	**Research questions/claims are presented clearly and accurately**
/15	**Proposed design and fabrication is presented clearly and accurately**
/25	**Analysis is comprehensive and rigorous, providing persuasive reasoning to support the research claims**
/25	**Figures and tables are clear, compelling, and support analysis.**
/20	**There is a clear storyline to the paper. It is thoughtfully organized and structured to argue its main points and claims. There is logical sequencing and logical transitions.**
/5	**Grammar and spelling, punctuation accurate**
/10	**No mechanics errors in the paper—figures/tables are accurately labeled and cited, etc.**
/5	**References cited correctly**
/15	**Report adheres to template guidelines**

Total: /145