Reviewer's comments are in black while our comments are in orange.

Please do note that the grammar and usage in the manuscript has been thoroughly reworked; the details of these changes are provided at the end of this rebuttal.

We are aware some of the notes in the protocol are exceedingly long. Unfortunately, these statements are important to the user of the protocol, representing subtle and hard-won fabrication facts that will help them avoid problems.

# Reviewer #1:

# Manuscript Summary:

Two fabrication techniques, lift-off and wet etching, are described to produce interdigital electrode transducers upon a piezoelectric substrate, lithium niobate, widely used to generate surface acoustic waves now finding broad utility in micro to nanoscale fluidics. The as-produced electrodes are shown to efficiently induce megahertz order Rayleigh surface acoustic waves.

# Major Concerns:

The manuscript explains two methods of fabricating IDT on piezoelectric substrate. The fabrication methods were explained briefly in several previous publications. I don't see any novelty in the current work.

Though the IDT fabrication process has been briefly explained in numerous publications from our and many other groups, the main benefit of this contribution is for those unfamiliar with the process. After having had many researchers contact us asking how to make these devices, with several researchers having visited our group in the past year alone just to learn how to make a SAW device, we believe there is a need to communicate a comprehensive protocol. We are not aware of any detailed video and written protocol describing the process, and in that sense we believe the work is novel, though we certainly agree with the reviewer that this is neither new science nor new engineering. We believe the details of the process are especially valuable, as perhaps nearly all researchers are unaware of black LN, the issue of birefringence when observing objects through LN, the importance of surface finish, and many other aspects we have sought to explain in this contribution.

However, this is the first manuscript details only the fabrication methods of IDT for applications related to acoustophoresis. The authors need to discuss the two methods in details before the manuscript is considered for publication. A missing part in the manuscript is the bonding of PDMS element to the piezoelectric substrate. Some publications suggest adding a very thin layer of PDMS on the substrate before the PDMS element is attached. Others are attaching the substrate and the PDMS element without adding the layer using O2-plasma. Modifying the protocol to cover the full fabrication method is essential.

Unlike the SAW IDT fabrication process, curiously, the PDMS bonding and surface processing protocols have been widely published in the literature, with Whitesides' group responsible for a majority of the publications on this topic. Notably, Friend and Yeo, Biomicrofluidics 2010 026502

is dedicated to this particular topic and may be beneficial to the reviewer. Further, because PDMS is really a terrible choice for acoustofluidics due to its extraordinary large acoustic loss, other choices are available in the literature such as thin film epoxy bonding in Langelier, et al., LoC 2012, 2970-2976, which is cited in our manuscript, and another forthcoming JoVE publication on direct LN-LN bonding for nanofluidics devices (#60648). That written, we certainly agree that PDMS is ubiquitous.

The problem with including other fabrication aspects is the format peculiar to JoVE does not permit lengthy elaboration on multiple fabrication topics. It is for this reason we have chosen to focus upon IDT fabrication via two closely related methods.

# Other comments include:

- 52- (normal photolithography) if this is a protocol then it's better to be specific.

"normal or" is deleted.

- Paragraphs from line 48 to 72 have no references.

Eleven additional citations have been added.

- 66- (The metal thickness should be 1% of the SAW wave length) needs more explanation.

It is re-explained with more detail.

- 69- The protocol is performed experimentally for only one frequency (100 MHz), more experiments with different parameters will make the protocol more beneficial.

It is true that only one frequency is described in the protocol. Changing the frequency will not affect many of the steps (or order) in fabricating the device. Only minor modifications will be necessary, and the risk is that the protocol will become convoluted and confusing as a consequence. Imagine stating something like " one would use AZ4562 for IDT structures with dimensions greater than 35  $\mu$ m, but AZ1518 for 20–35  $\mu$ m, AZ1512 for 15–25  $\mu$ m, and AZ1505 for 10–20  $\mu$ m"...and so on repeated for each photoresist and developer step. We chose to instead provide a concise protocol that would work as an introduction into the technique that could be readily adapted.

That written, a few sentences have been added explaining some of the basic design rules in the first paragraph of "Discussion" section as follows:

"Note that we focus upon producing a SAW device that resonates at 100 MHz in this protocol. If a different resonance frequency is required, the thickness of the sputtered metal film needs to be recalculated (1% of  $\lambda_{SAW}$ ). Also, the choice of photoresist needs to be adapted so that its thickness after spin-coating is at least twice that of the deposited metal. Further, the UV exposure and development times will need to be adjusted. It is generally possible to fabricate SAW devices that operate up to 700–800 MHz using these procedures with the appropriate

equipment, although in order to make a smooth pattern in microscale there are still some points worth discussing."

- The authors have not included any references that support the protocol being introduced. After line 47, there is no references for any information, equation, chemicals specifications ... etc.

11 more citations have been added.

- 184- usually this should be baking (or thermal treatment) instead of hard baking. The photoresist doesn't exist yet to call the step hard baking.

Changed to "Thermal treatment".

- 216- this caution should be applied for all chemical used not only at this step. Similar comments should be added for all other chemicals.

Agreed. Multiple "Cautions" are added to the protocol.

- While etching, I'm not sure the time is the only factor to decide if whether the sample etched properly or not. Observing the deposited material etched physically is more effective specially at micro and nano scale. Other factors might affect the etching process are temperature, etchant concentration, and newness.

Yes, higher temperature and concentration will reduce the time of etching. But the etching time is about 1-2 min at room temperature, which is still fairly short and easy to control. Shorter times introduce their own problems, mainly in the heterogeneity of etch across the wafer.

- 266- which software is used.

The equipment model and company name have been added to address this point, thank you.

- 271- more explanation about (mass loading effect) will be helpful.

Thank you. We have added more information to better explain the effect as follows:

"As the wavelength ( $\lambda_{SAW}$ ) is determined by the IDT design, the resonance frequency is usually a little lower than the designed value due to the effect of the mass loading of the metal electrode upon the substrate. This is a key reason why lighter metals, such as aluminum, tend to be used in higher frequency applications where this effect can be especially strong."

- 287- 0.7mm has a typo, it should be 6 to 7 mm. (according to ref. 10)

Changed to 7 mm.

- 311- travelling and standing can be generated. (it is better to write Surface Acoustic Waves)

Modified to "surface acoustic waves".

- A comparison between the two methods is going to be very beneficial.

A comparison of the two methods has been added to the "limitation" section.

#### TITLE:

2 Fabrication of Surface Acoustic Wave Devices on Lithium Niobate

3

1

#### **AUTHORS AND AFFILIATIONS:**

5 Jiyang Mei<sup>1</sup>, Naiqing Zhang<sup>1</sup>, James Friend<sup>1</sup>

6 7

<sup>1</sup>Medically Advanced Devices Laboratory, Center for Medical Devices and Instrumentation, Department of Mechanical and Aerospace Engineering, University of California San Diego, La Jolla, CA, USA

9 10 11

8

Corresponding author:

12 James Friend (jfriend@eng.ucsd.edu)

13

# 14 **KEYWORDS**:

Acoustofluidics, Surface Acoustic Waves, Lithium Niobate, Interdigital Transducers, Lift-Off, Wet Etching

19

20

21

## SUMMARY:

Two fabrication <u>technique</u>, lift-off and wet etching, are described <u>in producing</u> interdigital transducers, <u>which are widely used to generate surface acoustic waves on piezoelectric materials</u>, e.g., <u>lithium niobate</u>. <u>Both techniques</u> are shown to <u>produce useful 100</u> megahertz-order surface acoustic <u>wave devices</u>.

22 23 24

25

26

27

28

29 30

31

32

33

34

#### ABSTRACT:

Manipulation of fluids and particles by acoustic actuation at small scales has spurred rapid development of lab-on-a-chip applications. Megahertz-order surface acoustic wave (SAW) devices generate tremendous acceleration on their surface, up to  $10^8$  m/s², in turn responsible for many of the observed effects that have come to define acoustofluidics: acoustic streaming and acoustic radiation forces. These effects have been used for particle, cell, and fluid handling at the microscale, and even at the nanoscale. In this paper we explicitly demonstrate two major fabrication methods of SAW devices on lithium niobate: the details of lift-off and wet etching techniques are described step, by, step. Representative results for the electrode pattern deposited on the substrate and also the performance of SAW generated on the surface are provided in detail. Fabrication tricks and troubleshooting are covered as well. The aim is to provide the reader with a set of useful protocols for high frequency SAW device fabrication in future microfluidics applications.

39

40

41

42

43

44

# INTRODUCTION:

Relying on the well-known inverse piezoelectric effect, where the atomic dipoles create strain corresponding to the application of an electric field, piezoelectric single crystal media such as lithium niobate (LN) LiNbO3, and lithium tantalite (LT) LiTaO3, can be used as electromechanical transducers to generate SAW for microscale applications. For SAW is capable of producing surface displacements of up to 1 nm at high frequency, making it possible for SAW actuated acoustofluidics to overcome the obstacles of traditional ultrasonic methods: weak acceleration,

**Deleted:** techniques

Deleted: to produce

Deleted: electrode

Deleted: upon a piezoelectric substrate, lithium niobate,

**Deleted:** now finding broad utility in micro to nanoscale fluidics. The as-produced electrodes...

**Deleted:** efficiently induce

Deleted:

**Deleted:** Rayleigh

Deleted: waves

**Deleted:** is aiding the

Deleted: growth

Deleted: -

Deleted: -

Deleted: -

Deleted: displayed

**Deleted:** This procedure offers

Deleted: practical protocol

**Deleted:** and integration for

Deleted: crystals,

Deleted: ,

Deleted: ,

Deleted: applications1

**Deleted:** . By enabling the generation

**Deleted:** 10–1000 MHz, SAW-driven vibration overcomes the typical ...

**Deleted:** ultrasound: small

relatively long wavelengths, and difficulty in device miniaturization. Research to manipulate fluids and particles within small devices using SAW and related high frequency ultrasound has prospered in recent years as a consequence. 7-10

The fabrication of SAW-integrated microfluidic devices requires fabrication of the electrodes—the interdigital transducer (IDT)<sup>11</sup> on the piezoelectric substrate—to facilitate generating the SAW. The comb-shape fingers create compression and tension in the substrate when connected to an electric field; SAW is generated when this electric field is reversed at the resonance frequency of the SAW in the selected substrate. The fabrication process for SAW devices has been presented in many publications, whether using Jift-off ultraviolet photolithography alongside a metal sputter or a wet etching process. <sup>10</sup> For the lift-off technique, <sup>12-14</sup> a sacrificial layer (photoresist) with an inverse pattern is created on the surface, so that when the target material (metal) is deposited on the whole wafer, it can bond to the substrate in desired regions, followed by a "lift off" step to remove the remaining photoresist. By contrast, in the wet etching process, <sup>15-18</sup> the metal is first deposited on the wafer and photoresist is deposited and patterned on the metal, serving to protect the desired region from Jater being etched away by a metal etchant.

In the most commonly used design, the straight IDT, the wavelength of the resonance frequency of this SAW device is defined by the periodicity of the finger pairs, where the finger width and the spacing between fingers are both  $\lambda_{SAW}/4.^{19}$  In order to balance the electric current transmission efficiency and the mass loading effect on the substrate, the thickness of the metal deposited on the piezoelectric material is optimized to be about 1% of the SAW wavelength. 20 Localized heating from Ohmic losses, 21 potentially inducing premature finger failure, can occur if insufficient metal is deposited. A excessively thick metal film can cause a reduction in the resonance frequency of IDT due to a mass loading effect, and can possibly create unintentional acoustic cavities from the IDTs, isolating the acoustic waves they generate from the surrounding substrate. As a result, the photoresist and UV exposure parameters chosen vary in the lift-off technique, dependent principally upon the frequency but also the intended application for the device. Here, we describe in detail the lift-off process to fabricate a 100 MHz SAW-generating device on a double-sided polished 0.5 mm-thick 128° Y-rotated cut LN wafer, and the wet etching process to fabricate a 100 MHz device of identical design. Our approach enables one to consider these devices in investigation of a variety of physical problems and biological micro to nano-scale fluidics applications.

# PROTOCOL:

72 73

74

75

76

77

78

79

80

81

82

83

85

86

87

88

89 90

91

93

94

95

96

97

98

99

100

101

102

103

104

105

106 107

108

109 110

111

112

113

114 115

92

84

## 1. SAW Device Fabrication via Lift-Off Method

1.1. Wafer solvent cleaning: In a Class 100 clean room facility, immerse the 4" (101.6 mm) LN wafer into acetone, followed by isopropyl alcohol (IPA), and deionized water (DI water) in a sonication bath for 5 min, respectively. Pick up the wafer and use dry nitrogen (N<sub>2</sub>) gas flow over the surface of the wafer to remove the remaining DI water from the wafer.

CAUTION: Perform acetone and IPA immersion in a fume hood. Avoid inhalation and skin

Deleted: large

Deleted: ,

Deleted: large

Deleted: size.

Deleted: suspended

Deleted: recently accelerated, with a large number of

Deleted: and accessible reviews

Deleted: Fabrication

Deleted: mcirofluidic

Deleted: )-

Deleted:

Deleted: generate

Deleted: alternating

Deleted: input. The fabrication of

Deleted: normal or

Deleted: processes

**Deleted:** However, the lack of knowledge and skills in fabricating these devices is a key barrier to entry into acoustofluidics by many research groups, even today. ...

Deleted: reach

Deleted: created with a direct pattern

Deleted: "etching"

Deleted: a

Deleted: resonant

Deleted: depending

**Deleted:** different designs of SAW devices, especially

Deleted: as well as

Deleted: the

**Deleted:** offers a microfluidic system enabling

Deleted: then

Deleted: ), each

contact with IPA. Avoid skin and eye contact with acetone. Do not swallow.

Note: Do not allow any fluid to evaporate upon the wafer; if there is any dust or contamination on the surface, start this step over.

1.2. Pre-bake: Place the wafer onto a hotplate at 100 °C for 3 min.

147

148 149

150

151 152

153

154

155

156

157

158

159

160

161

162

163

164

165

166 167

168

169

170

171

172

173

174

175

176

177

178

179

180

181

182

183

184

185 186

187 188

189

190

NOTE: Because of the pyroelectric property of LN, it will generate static charges and associated stress within the wafer during heating and cooling. It is recommended to place the wafer onto a piece of aluminum (Al) foil after removing it from the hot plate to discharge any static charge from the wafer, thus preventing breakage.

1.3. Spin coating: Place the wafer onto a spin coater. Place negative photoresist (NR9-1500PY) onto the wafer using a dropper, covering about 75% of the wafer surface area. Program a speed of 500 rpm with acceleration of 3000 rpm/sec for 5 sec and then a speed of 3500 rpm with acceleration of 3000 rpm/sec for 40 sec, giving a thickness of the photoresist of around 1.3 μm.

CAUTION: Perform spin coating in a fume hood. Inhalation of photoresist fumes can cause headaches.

NOTE: The thickness may vary depending on the condition of the photoresist and the spin coater, even with the same rpm. After spin coating, the photoresist may be spun to the edge and over it to coat the back side edge of the wafer during spin coating. This must be removed by using a swab doused with acetone, If present, it will stick the wafer to the hotplate during the soft bake process, and the wafer will be difficult to pick up from the hotplate.

1.4. Soft bake: Place the wafer onto a hotplate at 25\_°C, ramp the temperature up to 150\_°C, hold it at 150 °C for 1 min, turn off the hotplate and let the wafer cool down to room temperature.

CAUTION: Avoid breathing in the photoresist. Inhalation of photoresist can cause headache.

NOTE: Due to the pyroelectric effect mentioned above, if the temperature of the LN wafer is suddenly changed, for example by directly transferring the LN wafer onto the hotplate or Al foil at 150\_°C, the sudden temperature change will cause thermal shock within the wafer, likely hattering it. The presence of nonuniform metal on the surface, such as electrodes, significantly, enhances the risk of wafer shattering due to differential stress. In applications where temperature excursions are necessary during fabrication or use, and the transparency of the LN is not important, consider using so-called "black" LN or more accurately reduced LN, which is a dark brown translucent color but more importantly has negligible pyroelectricity.

1.5. Ultraviolet exposure: Transfer the wafer to the mask aligner (MLA150). Expose the photoresist with an energy dose of 400 mJ/cm<sup>2</sup> at 375 nm to the wafer. The energy dose required may vary based on the mask design and the age and condition of the photoresist.

**Deleted:** debris remains

Formatted: Font: Not Italic

Deleted: put

**Deleted:** release the

Deleted: charges, and

Deleted: avoid breaking it

**Deleted:** Using

Deleted: cover

Deleted: wafer's

Deleted: with negative photoresist (NR9–1500PY).

Deleted: to produce

Deleted: thickness

Deleted: used

Deleted: settings. The

Deleted: beyond

Deleted: onto

Deleted: wafer's obverse

Deleted: ; this

Deleted: an

Deleted: -doused swab. Left

**Deleted:** the photoresist

Deleted: then

Deleted: and hotplate

Deleted: will

Deleted: shatter

Deleted: this

Deleted: .

Deleted: and

Deleted: Deleted: dose

Deleted: depending

NOTE: The wave propagation direction induced by IDTs should be along the X-propagating direction in order to effectively generate SAW. In other words, this implies the "fingers" of the IDT should be perpendicular to the X-axis direction. Typical LN wafer manufacturers place the primary (larger) wafer flat (straight edge alongside of wafer) perpendicular to the X-axis, so your IDT fingers should be parallel to this flat. Some manufacturers introduce a second (smaller) wafer flat to help indicate the Y and Z-axis directions, but this detail is unimportant for SAW generation. Manufacturers often request specifications for the surface finish of the wafer; if you require the ability to see through the wafer, request double-side optically polished wafers, though keep in mind that LN is birefringent, so any object illuminated with standard laboratory light and seen through the material will produce not one but two images.

generation if you do not need to see through the wafer, as spurious acoustic waves are diffused by the rough back surface.

1.6. Post-exposure bake: Place the wafer onto a hotplate at 100 °C for 3 min. Then transfer it onto Al foil to cool down to room temperature.

Overcoming this problem is discussed later. Single-side polished LN is a better choice for SAW

NOTE: The patterns should be able to be seen after the post-exposure bake. If not, consider stripping the photoresist and restarting the process from step 1.1 above.

1.7. Develop: Place the wafer into a beaker filled with pure RD6 developer for 15 sec. Gently shake the beaker during development. Immerse the wafer into DI water for 1 min, then rinse the wafer under DI water flow. Finally, use dry N<sub>2</sub> flow to remove the remaining DI water from the wafer. Never let any fluid evaporate on the wafer surface.

<u>CAUTION: Develop the wafer in a fume hood. Avoid breathing vapors or developer contact with</u> eyes and skin.

NOTE: The photolithography is complete at this step. The protocol can be paused here.

1.8. Hard bake: Place the wafer onto a hotplate at 100  $^{\circ}$ C for 3 min. Then transfer it onto Al foil to cool <u>down</u> to room temperature.

Note: This step is to remove any moisture on the wafer preventing outgassing during sputtering that could reduce the quality of the deposited metal film.

1.9. Electrode sputter deposition: Place the wafer into a sputter deposition system. Vacuum the chamber to  $5 \times 10^6$  mTorr. Use argon flow at 2.5 mTorr, sputter Cr with a power of  $\underline{200 \text{ W}}$  for 5 nm as an adhesion layer, followed by sputtering Al with a power of 300 W for 400 nm to form the conductive electrodes.

NOTE: Deposition time should be calculated from the expected thickness and the deposition rate. Titanium (Ti) can be used instead of chromium, though the process is more difficult as Ti is

**Deleted:** and allow it

**Deleted:** after

Deleted: from

**Deleted:** and photoresist to prevent later

Deleted: 200W

270 tougher. For higher frequency SAW devices, Al should replace Au to avoid the mass loading 271 effects of the Au IDT fingers which reduce the local SAW resonance frequency under the IDT, 272 forming an acoustic cavity from which the SAW can only escape with significant loss. 273 274 1.10. Lift-off\_process: Transfer the wafer into a beaker and immerse into acetone. Sonicate at Deleted: immersed with 275 medium intensity for 5 min. Rinse with DI water and dry the wafer with dry N<sub>2</sub> flow. 276 Formatted: Normal (Web) 277 CAUTION: Use acetone in a fume hood. Avoid inhalation and skin or eye contact with acetone. 278 Do not swallow. 279 280 1.11. Dicing process: Use a dicing saw to dice the entire wafer into small pieces of chips as SAW 281 devices for further applications. 282 283 NOTE: The fabrication is complete. The protocol can be paused here. 284 285 NOTE: Instead of a saw, a diamond-tipped wafer scribe (or even a glass cutter) can be used to 286 dice the LN wafer with some practice, though due to the anisotropy of LN it is important to scribe and break the wafer first along scribe lines perpendicular to the X-axis, followed by those 287 288 lines along the X-axis. 289 2. SAW Device Fabrication via Wet Etching Method 290 291 292 2.1. Wafer solvent cleaning: In a Class 100 clean room facility, immerse the 4" (101.6 mm) LN 293 wafer into acetone, followed by IPA, and deionized DI water in a sonication bath for 5 min, 294 respectively. Pick up the wafer and use dry N2 gas flow over the surface of the wafer to remove 295 the remaining DI water from the wafer. 296 297 CAUTION: Use acetone and IPA in a fume hood. Avoid inhalation and skin contact with IPA. 298 Avoid acetone contact with skin and eyes. Do not swallow. 299 300 Note: Do not allow any fluid to evaporate upon the wafer; if there is any dust or contamination Deleted: debris 301 on the surface, start this step over. 302 303 2.2. Thermal treatment: Place the wafer onto a hotplate at 100 °C for 3 min. Then transfer it Deleted: Hard bake 304 onto Al foil to cool down to room temperature. 305 306 2.3. Electrode sputter deposition: Place the wafer into a sputter deposition system. Vacuum 307 the chamber to  $5 \times 10^{-6}$  mTorr. Use argon flow at 2.5 mTorr, sputter Cr with a power of 200 W 308 for 5 nm as an adhesion layer, followed by sputtering Au with a power of 300 W for 400 nm to 309 form the conductive electrodes. 310 311 NOTE: The protocol can be paused here. 312 2.4. Spin coating: Place the wafer onto a spin coater. Place positive photoresist (AZ1512) onto Deleted: Using 313

the wafer using a dropper, covering about 75% of the wafer surface area. Program a speed of 500 rpm with acceleration of 3000 rpm/sec for 10 sec and then a speed of 4000 rpm with acceleration of 3000 rpm/sec for 30 sec, giving a thickness of the photoresist at around 1.2 μm.

318

319

320

321

322

323

324 325

326

327

328

329

330

331

332

333

334

335

336

337

338

339

340 341

342

343

344

345

346

347

348

349

350

351

352 353

354

355

356 357

358

359 360

361

<u>CAUTION: Perform spin coating in a fume hood. Inhalation of photoresist fumes can cause headaches.</u>

NOTE: The thickness may vary depending on the condition of the photoresist and the <u>model of</u> spin coater used, even <u>at</u> the same <u>rpm. After spin coating, the</u> photoresist may be spun <u>to</u> the edge and <u>over it to coat</u> the <u>back side</u> edge of the <u>wafer. This</u> must be removed by using <u>a swab doused with</u> acetone. <u>If</u> present, <u>it</u> will stick the wafer to the hotplate during the soft bake <u>process, and the wafer will be difficult to pick up from the hotplate</u>.

2.5. Soft bake: Place the wafer onto a hotplate at 100 °C for 3 min. Then transfer it onto Al foil to cool down to room temperature.

2.6. <u>Ultraviolet exposure:</u> Transfer the wafer to the mask aligner (MLA150). Expose the photoresist with an energy dose of 150 mJ/cm<sup>2</sup> at 375 nm, to the wafer. The energy dose required may vary <u>based</u> on the mask design and the age and condition of the photoresist.

2.7. Develop: Place the wafer into a beaker filled with pure AZ300MIF developer for 30 sec. Gently shake the beaker during development. Immerse the wafer into DI water for 1 min, then rinse the wafer under DI water flow. Finally, use dry  $N_2$  flow to remove the remaining DI water from the wafer. Never let any fluid evaporate on the wafer surface.

CAUTION: Avoid <u>AZ300MIF</u> contact with <u>skin or eyes</u>. Do not swallow.

2.8. Au etching: Immerse the wafer into a beaker filled with Au etchant for 90 sec, gently shaking the beaker. After <u>rinsing</u> the wafer under DI water flow, use dry N<sub>2</sub> flow to remove the remaining DI water from the wafer. Never let any fluid evaporate on the wafer surface.

CAUTION: Gold etchant can be hazardous <u>for</u> the eyes and skin, and <u>causes</u> respiratory irritation. This step requires higher level personal protective equipment (PPE), such as safety <u>glasses</u>, black neoprene gloves, apron, etc.

2.9. Cr etching: Immerse the wafer into a beaker filled with Cr etchant for 20 sec, gently shaking the beaker. After rinsing the wafer under DI water flow, use dry  $N_2$  flow to remove the remaining DI water from the wafer. Never let any fluid evaporate on the wafer surface.

CAUTION: Chromium etchant can cause eye, skin and respiratory irritation. This step also requires higher level PPE.

2.10. Sample cleaning: Put the wafer into acetone, followed by IPA, and DI water in a sonication bath for 5 min, respectively. Pick up the wafer and use dry N2 gas flow over the surface of the

Deleted: cover

Deleted: wafer's

Deleted: with positive photoresist (AZ1512).

Deleted: ultimately producing

Deleted: laver of

Deleted: with

Deleted: settings. The

Deleted: beyond

Deleted: onto

Deleted: wafer's obverse

Deleted: ; this

Deleted: -doused swab. Left

**Deleted:** the photoresist

Deleted: .

Deleted: dose

Deleted: depending

**Deleted:** skin or eye

Deleted: AZ300MIF

Deleted: rinse

Deleted: to

Deleted: will cause

Deleted: glass

wafer to remove the remaining DI water from the wafer.

<u>CAUTION: Use acetone in a fume hood. Avoid inhalation and skin contact acetone with skin and eyes. Do not swallow.</u>

NOTE: This step is to remove undesired photoresist <u>from</u> the wafer.

2.11. Dicing process: Use a dicing saw to dice the entire wafer into chips as SAW devices for further use.

NOTE: The fabrication is completed. The protocol can be paused here.

#### **Experimental Setup and Testing**

385

386 387

388

389 390

391 392

393 394

395

396 397

398

399 400

401

402

403

404

405

406

407

408

409

410

411

412

413

414

415

416

417

418

419

420

421

422

423

424

425

426

427

428

1. Observation: Observe the SAW device under an optical microscope in bright field mode.

NOTE: <u>Scratches may be present upon</u> the metal layers <u>deposited</u> on the LN. Generally, they will not cause a notable influence of the device performance, as long as the scratches <u>do</u> not result in an open circuit.

2. SAW actuation: Attach absorbers at both ends along the propagation direction of the SAW device to prevent reflected acoustic waves from the edges. Use a signal generator to apply a sinusoidal electric field to the IDT at its resonance frequency around 100 MHz. An amplifier may be connected to amplify the signal. Use an oscilloscope to measure the actual voltage, current, and power applied to the device. The amplitude and frequency response of the SAW generated by the device may be measured by a laser Doppler vibrometer (LDV\_UHF-120, Polytec, Waldbronn, Germany). The SAW-actuated droplet motion may be recorded using a high-speed camera attached to the microscope.

# **REPRESENTATIVE RESULTS:**

The IDT to be measured is designed to have a resonance frequency of 100 MHz, as the spacing between the fingers of the IDT and the widths of the fingers themselves are all 10 µm, making the wavelength 40 µm. Figure 1(B) is an image of the IDTs fabricated using the method described above.

Applying an sinusoidal signal to the JDT near the designed resonance frequency allows SAW to be generated to propagate across the surface of the piezoelectric material. The LDV measures the vibration via the Doppler effect on the surface, and through signal processing, information such as amplitude, velocity, acceleration, and phase can be acquired and displayed. We illustrate the frequency response under a frequency sweep from 90 to 105 MHz using an input power of 140 mW, a peak-to-peak voltage of 70 V, and peak-to-peak current of 720 mA. As Figure 2(A) indicates, the resonance frequency is measured to be 96.5844 MHz, slightly lower than the design frequency of 100 MHz, attributable to the mass loading of the deposited metal, for an amplitude of 19.444 pm. Figure 2(B) plots the vibration upon the substrate surface, showing a SAW to be propagating from the IDTs. The standing wave ratio (SWR) is calculated to

**Deleted:** the undesired photoresist on

Deleted: discrete

Formatted: Normal (Web)

**Deleted:** - field optical microscopy

... [2]

.. [1]

**Deleted:** There are possibly scratches across...cratches may be present upon the metal layers deposited on the LN. Generally, they will not cause a notable influence of the device performance, as long as the scratches are...o not deep enough to result in a .... [3]

Deleted: of around 100 MHz. An amplifier should...ay be connected to amplify the signal. Use an oscilloscope to measure the actual voltage, current, and power applied ontot the device. The amplitude and frequency response of of the SAW are...enerated by the device may be measured by a laser Doppler vibrometer (LDV); the ... UHF-120, Polytec, Waldbronn, Germany). The SAW-actuated droplet motion [4]

**Deleted:** atof 100 MHz, as the finger width and the spacing between them...he fingers of the IDT and the widths of the fingers themselves are all  $10~\mu m$ , producing amaking the wavelength of ...0  $\mu m$ . Figure 1(B) shows...s an IDT image of the IDTs fabricated using this...

Deleted: Using...pplying an oscillating electrical...inusoidal signal matched ... o the resonant...DT near the designed resonance frequency of the IDT,...llows SAW can...o be generated to propagate across the surface of the piezoelectric material. The LDV measures the vibration via the Doppler effect on the surface, and through signal processing, information such as amplitude, velocity. acceleration, and phase can be acquired and displayed using the software.. We illustrate the frequency response under a frequency sweep from 90 to 105 MHz, with...using an input power of 140 mW, a peak-to-peak voltage of 70 V, ...nd a peak-to-peak current of 720 mA. As Figure 2(A) indicates, the amplitude of the SAW is 19,444 pm at a ...esonance frequency of ...s measured to be 96.5844 MHz. The slight reduction in ... slightly lower than the design frequency from theof 100 MHz design is attributed... attributable to the mass mass loading of the deposited metal IDT electrodes.... for an amplitude of 19.444 pm. Figure 2(B) illustrates...lots →the LDV-measured ...ibration of...pon the SAW on the...ubstrate surface, shown ...

be  $2.06_{\underline{e}}$  from the ratio of maximum amplitude to minimum amplitude, A SWR = 1 is produced for a pure traveling wave while SWR =  $\infty$  for a pure standing wave, suggesting the value of SWR =  $2.06_{\underline{e}}$  is a good traveling wave,

We also demonstrate the motion of a sessile droplet actuated by the SAW device while using a single frequency signal input (80.6 mW) at its resonance (96.5844 MHz). A <u>water</u> droplet of 0.2 <u>uL</u> is pipetted on LN about 1 mm away from the IDT (see Figure 3(A)). When SAW propagates and encounters the droplet <u>(water)</u> on the surface, it "leaks" into the liquid at the Rayleigh angle, <u>itself determined by</u> the ratio of sound <u>speeds between sound</u> in <u>the water and SAW upon the substrate</u>,

$$\theta_R = \sin^{-1}\left(\frac{v_{water}}{v_{LN}}\right) = \sin^{-1}\left(\frac{1498 \, m/s}{3992 \, m/s}\right) \approx 22^{\circ}.$$

The jetting angle shown in Figure 3(B) confirms the presence of SAW.

## FIGURE AND TABLE LEGENDS:

515

516

517

518

519

520

521

522

523

524

525

526 527

528

529

530

531

532

533

534

535

536

537

538

539

540

541

542

543

544

545

546

547

548

549

550

551

552

553

554

555

556

557

Figure 1: Images of fabricated devices. (A) <u>Gold</u>-electrode <u>DTs</u> with 7 mm aperture on an LN substrate for 100 MHz SAW generation and propagation. (B) The <u>IDT</u> fingers <u>upon</u> the <u>LN substrate</u>; note the gratings on the left <u>which serve as</u> reflectors to prevent <u>spurious SAW back</u> reflection from the chip edge. These gratings have similar design features to the IDT fingers but are not connected to each other in this version. Scale bar: 200 µm. The inset illustrates the details of the fingers at greater magnification. Scale bar: 50 µm.

**Figure 2: LDV measurement of the SAW device.** (A) The frequency response (amplitude vs frequency) from 90 MHz to 105 MHz<u>indicating the presence of a</u> resonance at 96.5844 MHz with 19.444 pm amplitude. (B) A snapshot of the traveling wave generated by the IDT at the <u>resonance frequency as it propagates across</u> the LN substrate <u>surface</u>.

Figure 3: SAW-induced droplet jetting. (A) The experimental setup for SAW-induced sessile drop actuation on LN. Scale bar: 5 mm. (B) SAW is propagating from the left to right in the images, at 80.6 mW power input, inducing the droplet jetting as shown. The angle is measured to be around the Rayleigh angle (22°). Scale bar: 1 mm.

Figure 4: Scheme for photoresist left on the substrate. (A) When positive photoresist is used, it is left in a trapezoidal shape with positively sloped edges after the development step. Metal deposited upon this structure will be difficult to remove. (B) When negative photoresist is used, however, this trapezoidal shape is inverted, with significant overhang that makes lift-off of the deposited metal easier in a later step.

# **DISCUSSION:**

Whichever method is chosen, it is possible to make SAW devices capable of generating good surface acoustic waves. Due to the deposition of metal in forming the IDTs, a small amount of additional mass is present upon the surface, reducing the SAW velocity in this region. As the wavelength ( $\lambda_{\rm SAW}$ ) is determined by the IDT design, the resonance frequency is usually a little lower than the designed value due to the effect of the mass loading of the metal electrode

**Deleted:** , determined by using...from the ratio of maximum amplitude to the ...inimum amplitude (...

Deleted: ),

Deleted: has been obtained here

Deleted: , under while using a single frequency signal input input (80.6 mW) at its resonance (96.5844 MHz). A 0.2 μL...ater droplet of 0.2 μL is pipetted on LN about 1 mm away from the IDT (see Figure 3(A)). When SAW propagates and encounters the water ...roplet upon... [8]

Formatted: Font: Not Italic

 $\begin{tabular}{ll} \textbf{Deleted:} because of the impedance difference from LN to water, and calculated as...tself determined by the ratio <math>\begin{tabular}{ll} \begin{tabular}{ll} \begin{$ 

**Deleted:**  $\sin^{-1}\left(\frac{v_{water}}{v_{LN,SAW}}\right)$ 

Deleted: A gold...old-electrode IDT...DTs with 0.... mm aperture on an LN substrate for 100 MHz SAW generation and propagation. (B) The IDT fingers of...pon the IDTs.... [10]

Moved down [1]: . Scale bar: 200 μm.

Deleted: (The gratings on the left are

Moved (insertion) [1]

**Deleted:** energy loss.)

**Deleted:** of the SAW device from 90 MHz to 105 MHz includes its... indicating the presence of a resonance at 96.5844 MHz with 19.444 pm amplitude. (B) A snapshot of the traveling wave generated by the IDT at the input level of 1 V from the LDV. (B) The SAW present upon...esonance frequency as it propagates across the LN substrate as it propagates from the IDT. The phase has been determined by scanning the LDV head to measure in multiple locations, with the phase referenced against the input electrical signals [12]

**Deleted:** . The ...at 80.6 mW power input, inducing the droplet jetting, at approximately...as shown. The angle is measured to be around the Rayleigh angle (22°) occurs at 80.6 mW power input.°

Formatted: Font: Not Italic

**Deleted:** , making it far that makes lift-off of the deposited deposited metal easier to dissolve the underlying photoresist and remove the metal during liftoffi

Deleted: SAW devices fabricated from either method are capable of generating useful traveling waves on the surface, and these methods underpin more complex processes to produce other designs. The resonance frequency is usually a little lower than the designed value, due to the mass loading effect of the metal deposited on top. However, there are 1818

f · ... [7]

[11]

upon the substrate. This is a key reason why lighter metals, such as aluminum, tend to be used in higher frequency applications where this effect can be especially strong. Note that we focus upon producing a SAW device that resonates at 100 MHz in this protocol. If a different resonance frequency is required, the thickness of the sputtered metal film needs to be recalculated (1% of  $\lambda_{\text{SAW}}$ ). Also, the choice of photoresist needs to be adapted so that its thickness after spin-coating is at least twice that of the deposited metal. Further, the UV exposure and development times will need to be adjusted. It is generally possible to fabricate SAW devices that operate up to 700–800 MHz using these procedures with the appropriate equipment, although in order to make a smooth pattern in microscale there are still some points worth discussing.

## 1. Lift-off Method

662

663

664

665

666

667

668

669

670

671

672

673

674

675

676

677

678

679 680

681

682

683

684

685

686

687

688

689

690

691

692

693

694

695

696

697

698

699

700

701

702

703

704

705

The first aspect to consider is the choice of photoresist, Typically, negative photoresists are used for lift-off fabrication, though they tend to be more difficult to work with than positive photoresists, as they tend to be more difficult to strip off in the final step and can exhibit deswelling. The regions of the positive photoresist exposed to UV will be dissolved, leaving behind the unexposed regions. The boundaries between these regions form almost inevitably form a trapezoidal cross-section with sloped edges, especially when underexposed, shown with exaggeration for clarity in Figure 4(A). The metal sputtered atop the photoresist in this shape will prevent the developing solvent from penetrating and dissolving the photoresist at the corners of the trapezoidal shape nearest the substrate, making it difficult to remove the metal in the lift-off step. On the other hand, Figure 4(B) is an example when using a negative photoresist, where regions unexposed to UV are dissolved by the developer. The trapezoidal shape of the cross section still tends to form on the substrate, but inverted with overhang, making the lift-off much easier. Apart from the lift-off problem when using a positive photoresist, the fingers will eventually be thinner and spaced slightly wider than designed due to the trapezoidal profile. Conversely, they will be wider and more narrowly spaced when using a negative photoresist.

Assuming the use of a negative photoresist, the <u>UV</u> exposure dose <u>will also be crucial in the fabrication</u>. Due to the <u>different</u> equipment and reagent used, the exposure time <u>may still vary.</u> If it is over-exposed, meaning the exposed area is larger than wanted, which will cause the fingers to be narrower and the spacing wider than designed. <u>Oppositely, if it is under-exposed, there is possibly</u> some of the photoresist <u>left</u> after development, in which case the metal in the desired area will peel off together with the thin layer of the remaining photoresist after lift-off. Sometimes people tend to use a single polished LN wafer, as mentioned above, which is opalescent. The time/dose of <u>UV</u> exposure required for such <u>wafers</u> will be increased, since the light <u>will be</u> diffused at the back.

# 2. Wet Etching Method

The key step for this method is <u>that make sure that</u> the photoresist is completely dissolved <u>for</u> the area <u>of</u> metal <u>that</u> needs to be etched away, otherwise the etchant will be blocked.

As the process of the metal etching is isotropic, suggesting the etching not only occurs

Deleted: is important. It is possible to use a

**Deleted:** for the fabrication, which, nevertheless, will be more difficult. Because...

**Deleted:** photoresist is dissolved, the part left on the substrate willr

Deleted: shape

**Deleted:** with underexposure, as exaggerated

Deleted: on

Deleted: top of such a

Deleted: result in difficulties in removing it during

Deleted: UV-exposed regions of

Deleted: removed, and, as shown in Figure 4(B), an

Deleted: trapezoid is typically formed

Deleted: that makes

Deleted: step

Deleted: ¶

Deleted: of

**Deleted:** , i.e., the spacing between them will be slightly larger ...

Deleted: shape. With

**Deleted:** spacing is smaller. These effects slightly change the resonance frequency from the design intent. ¶

When using negative photoresist, the UV

Deleted: is crucially important.

Deleted: variety of

**Deleted:**, photoresists, and reagents available today

**Deleted:** required in your fabrication process will very likely vary. Observation of the fabricated device result can guide you in trying to determine what went wrong. Over 171

**Deleted:** Under-exposure may leave

Deleted: and

Deleted: UV exposure with

Deleted: a wafer

Deleted: is

Deleted: to ensure

**Deleted:** from

Deleted: where

Deleted: and

Deleted: lithography will fail. ¶

... [18

Deleted: it

perpendicularly, but also across the metal layer, the metal left behind—in this case as IDT fingers will be narrower than expected. Negative photoresist is therefore a better choice in this technique so as to compensate for the undesired feature loss.

#### 3. Limitations

755

756

757

758

759

760

761

762

763

764

765

766

767

768 769

770

771

772

773

774

775 776

777

778 779

780

781

782

783

784

785

787

One of the advantages of the lift-off method over wet etching is that it achieves reproducibly defined structures that can always be easily washed away to restart the process if a problem is found. Both methods have a limit (e.g. isotropic etching) in the resolution of the structures they can reliably reproduce at about a few micrometers. According to our experience in our facilities, the practical limit is around 2-3 µm. If sub-micron features are required, other fabrication techniques may be called upon.

## **ACKNOWLEDGMENTS:**

The authors are grateful to the University of California and the NANO3 facility at UC San Diego for provision of funds and facilities in support of this work. This work was performed in part at the San Diego Nanotechnology Infrastructure (SDNI) of UCSD, a member of the National Nanotechnology Coordinated Infrastructure, which is supported by the National Science Foundation (Grant ECCS-1542148). The work presented here was generously supported by a research grant from the W.M. Keck Foundation. The authors are also grateful for the support of this work by the Office of Naval Research (via Grant 12368098).

#### **DISCLOSURES:**

The authors have nothing to disclose.

### **REFERENCES:**

[1] Ding, X., Lin, S. C. S., Lapsley, M. I., Li, S., Guo, X., Chan, C. Y., & Huang, T. J. (2012). Standing surface acoustic wave (SSAW) based multichannel cell sorting. Lab on a Chip, 12(21), 4228-4231. [2] Langelier, S. M., Yeo, L. Y., & Friend, J. (2012). UV epoxy bonding for enhanced SAW transmission and microscale acoustofluidic integration. Lab on a Chip, 12(16), 2970-2976.

[3] Rezk, A. R., Qi, A., Friend, J. R., Li, W. H., & Yeo, L. Y. (2012). Uniform mixing in paper-based microfluidic systems using surface acoustic waves. Lab on a Chip, 12(4), 773-779.

786 [4] Schmid, L., Weitz, D. A., & Franke, T. (2014). Sorting drops and cells with acoustics: acoustic microfluidic fluorescence-activated cell sorter. Lab on a Chip, 14(19), 3710-3718.

788 [5] Schmid, L., Wixforth, A., Weitz, D. A., & Franke, T. (2012). Novel surface acoustic wave 789 (SAW)-driven closed PDMS flow chamber. Microfluidics and nanofluidics, 12(1-4), 229-235.

790 [6] Shi, J., Mao, X., Ahmed, D., Colletti, A., & Huang, T. J. (2008). Focusing microparticles in a 791 microfluidic channel with standing surface acoustic waves (SSAW). Lab on a Chip, 8(2), 221-223.

792 [7] Friend, J., & Yeo, L. Y. (2011). Microscale acoustofluidics: Microfluidics driven via acoustics 793 and ultrasonics. Reviews of Modern Physics, 83(2), 647.

794 [8] Ding, X., et al. (2013). Surface acoustic wave microfluidics. Lab on a Chip, 13(18), 3626-3649.

795 [9] Destgeer, G., & Sung, H. J. (2015). Recent advances in microfluidic actuation and micro-796 object manipulation via surface acoustic waves. Lab on a Chip, 15(13), 2722-2738.

797 [10] Connacher, W., et al. (2018). Micro/nano acoustofluidics: materials, phenomena, design, 798 devices, and applications. Lab on a Chip, 18(14), 1952-1996.

Deleted: both through and

Deleted: making the

**Deleted:** designed

Deleted: reduce

Deleted: are limited to fabricating feature sizes to greater

Deleted: can be pushed to as small as

Deleted: submicron

- 807 [11] White, R. M., & Voltmer, F. W. (1965). Direct piezoelectric coupling to surface elastic
- 808 waves. Applied physics letters, 7(12), 314-316.
- [12] Smith, H. I., Bachner, F. J., & Efremow, N. (1971). A High-Yield Photolithographic Technique
- 810 <u>for Surface Wave Devices. Journal of the Electrochemical Society, 118(5), 821-825.</u>
- 811 [13] Bahr, A. (1973, September). Fabrication techniques for surface-acoustic-wave devices.
- 812 In Proc. Int. Specialists Seminar on Component Performance and Systems Applications of
- 813 Surface Acoustic Wave Devices.
- [14] Smith, H. I. (1974). Fabrication techniques for surface-acoustic-wave and thin-film optical
- 815 <u>devices. Proceedings of the IEEE, 62(10), 1361-1387.</u>
- 816 [15] Wilke, N., Mulcahy, A., Ye, S. R., & Morrissey, A. (2005). Process optimization and
- 817 <u>characterization of silicon microneedles fabricated by wet etch technology. Microelectronics</u>
- 818 <u>Journal, 36(7), 650-656.</u>
- [16] Madou, M. J. (2002). Fundamentals of microfabrication: the science of miniaturization. CRC
- 820 press.
- [17] Köhler, M. Etching in Microsystem Technology. 1999.
- 822 [18] Brodie, I., & Muray, J. J. (2013). The physics of micro/nano-fabrication. Springer Science &
- 823 Business Media.
- 824 [19] Dentry, M. B., Yeo, L. Y., & Friend, J. R. (2014). Frequency effects on the scale and behavior
- of acoustic streaming. Physical Review E, 89(1), 013203.
- 826 [20] Morgan, D. (2010). Surface acoustic wave filters: With applications to electronic
- 827 <u>communications and signal processing. Academic Press.</u>
- 828 [21] Pekarcikova, M., Hofmann, M., Menzel, S., Schrnidt, H., Gemming, T., & Wetzig, K. (2005).
- 829 Investigation of high power effects on Ti/Al and Ta-Si-N/Cu/Ta-Si-N electrodes for SAW
- devices. IEEE transactions on ultrasonics, ferroelectrics, and frequency control, 52(5), 911-917.