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Dear Sir/Madam:

We prepared for the revised manuscript again. We are grateful to you if the manuscript is reviewed again.

TITLE:

Enhancement Method of Surface Acoustic Wave-Atomizer Efficiency for Olfactory Display

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KEYWORDS:

21 SAW atomizer, olfactory display, amorphous Teflon coating, smell persistence, micro-dispenser, 22 silanization, dip coating

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SUMMARY:

We establish here a method for coating the surface of a surface acoustic wave (SAW) device with amorphous Teflon film to improve the atomization efficiency required for application to an olfactory display.

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ABSTRACT:

Since olfaction is an important sense in human interfaces, we have developed an olfactory display using a surface acoustic wave (SAW) atomizer and micro-dispensers. In this olfactory display, the efficiency of atomization is important in order to avoid smell persistence problems often encountered in human olfactory interfaces. Thus, the SAW device is coated with amorphous Teflon film to change the substrate nature from hydrophilic to hydrophobic. It is also necessary to silanize the piezoelectric substrate surface prior to Teflon coating to enhance the adhesion of the film. A dip coating method was adopted to obtain uniform coating on the substrate. The highspeed solenoid valve was used as micro-dispenser to spout a liquid droplet to the SAW device surface since its accuracy and reproducibility were high. Then, the atomization became easier on the hydrophobic substrate. In this study, the amorphous Teflon coating for minimizing the remaining liquid on the substrate after atomization was studied. The goal of the protocol described here is to show the methods for coating a SAW device surface with amorphous Teflon film and generating the smell using the SAW atomizer and a micro-dispenser, followed by performance of a sensory test.

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INTRODUCTION:

Although devices for stimulating visual and auditory senses are popular, we cannot present all sensations that we perceive; though, we can ordinarily present a sensation using only these two senses. An olfactory display is a gadget that can present a scent, and it is used in virtual reality so that a user can perceive scents¹⁻⁷. Since olfaction contributes to emotions greatly, an olfactory stimulus is indispensable for enhancing reality. We have previously studied movies, animations, and games with scents^{8,9}.

Several researchers have studied olfactory displays; for example, Yanagida has studied a scent projector that delivers a scent to a specified person even when no one around him or her perceives it¹. Yamada et al. have studied an odor source localization in virtual space using a simple Gaussian distribution model of odor concentration². Kim et al. have proposed the concept of two-dimensional arrays of odor-releasing devices ³. Moreover, simple wearable olfactory displays and the ultrasonic phased array for controlling the direction of these scents have been proposed⁴⁻⁶.

One of the problems in olfactory display is smell persistence. A user may detect the smell even after it is intended to be changed to the air or another scent. Since it is preferable to switch between scents as quickly as possible in virtual reality, the smell persistence problem should be studied.

 We have studied the olfactory display with a function of blending many ingredients. We previously developed this system using solenoid valves with high-speed switching¹⁰. Although it stably blends many ingredients, we could not yet solve the problem of smell persistence. We have since developed the olfactory display using micro-dispensers and a SAW atomizer¹¹. Although similar techniques have been used to manipulate liquid droplets¹²⁻¹⁴, we applied it to scent generation. The SAW device is suitable for atomizing liquid droplets since it can atomize liquid droplets instantaneously^{15,16}; however, we have found that tiny liquid droplets stay on a piezoelectric substrate after the atomization. These tiny liquid droplets cause smell persistence, even if most of the liquid is atomized.

Typically, a perfume is dissolved into a solvent such as ethanol to reduce viscosity. However, diluted perfume spreads onto the surface of a piezoelectric substrate due to its hydrophilic nature, and the atomization efficiency deteriorates when thin film spreads. Thus, a part of the liquid remains even after atomization, which cannot be removed even if RF power increases. Since the solvent evaporates soon after, only the perfume remains on and sticks to the substrate.

In this study, we coat the surface of a piezoelectric substrate with thin amorphous Teflon film so it becomes hydrophobic in nature. Since we can keep the droplet-like sphere on the hydrophobic surface, the energy required for detaching liquid from the substrate surface decreases. It is expected that an atomization efficiency is improved when the surface of SAW device becomes hydrophobic. The overall goal of this method is to improve the atomization efficiency so that a scent is immediately presented and can quickly disappear after its presentation, ultimately for application to olfactory display. In this paper, we show how a SAW device is coated with amorphous Teflon film and demonstrate the improvement of atomization efficiency and its

experimental results were described in the reference¹⁷.

PROTOCOL:

The methods described here have been approved by the Human Research Ethics Committee of Tokyo Institute of Technology.

1. SAW Device Preparation and Checking Impedance

1.1 Prepare a 10 MHz SAW device on a LiNbO₃ substrate [128°-rotated Y-cut, X propagation, with 21 finger pairs of an IDT (inter-digitated transducer)], together with reflectors made of 32 finger pairs on one IDT side, as shown in **Figure 1a**.

Note: **Figure 1b** shows the principle of atomization. The SAW is converted to a longitudinal wave at the liquid droplet. Mist is generated due to acoustic streaming if the SAW energy is sufficiently large.

Note: The SAW device was fabricated by the manufacturer using typical photo lithography according to the electrode pattern that the authors designed. The piezoelectric substrate mentioned above was selected due to a high electromechanical coupling coefficient.

1.2 Mount the SAW device using aluminum foil and conductive paste on a printed circuit board made of aluminum (designed for this SAW device) where the SMA connector is attached (Figure 2).

113 Note: A printed circuit board made of aluminum is effective for heat radiation.

1.3 Measure the frequency characteristic of impedance using a network analyzer. The SAW device is connected to the analyzer via a coaxial cable connection from the printed circuit board. Frequency characteristics should show areas where imaginary parts of the device admittance go to zero, which are the SAW device oscillation frequencies.

Note: When acoustic loss in the device is large, atomization does not occur. The acoustic loss can be checked by measuring the frequency characteristic of impedance. When the SAW device is coated with Teflon film, the difference between the frequency characteristics before and after coating should be monitored to check if the film is too thick.

2. Silanization

2.1 Prepare the amino-based silane coupling agent (3-ahhminopropyltriethoxy silane). Adjust its concentration to 0.5 % (v/v) in water using a pipette.

Note: Silanization is mandatory to enhance the adhesion of amorphous Teflon coating. Teflon coating is removed during atomization if silanization is not performed.

133	2.2	Clean the surface of the SAW device using a cotton swab soaked with acetone.
134	2.2	Call the de land a disconder (Fig. 12.2)
135 136	2.3	Set the device at a dip coater (Figure 3).
137 138 139 140	the dip	A printed circuit board where the SAW device is fixed using mending tape is attached to coater, since the thickness of the SAW device (0.5 mm) is too thin to be attached to the atter directly.
141	2.4	Pull down the device so that the atomization area can be immersed in the solution at a
142	<mark>speed</mark>	of 0.2 mm/s. Keep the device in solution for 5 min.
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144	2.5	Pull up the device at a speed of 1.7 mm/s. Keep the device in the air for 5 min.
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146	<mark>2.6</mark>	Rinse the device in pure water for 1 min.
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148	2.7	Keep the device in the air for 30 min.
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150	3. An	norphous Teflon Coating
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152	3.1	Prepare the amorphous Teflon material and solvent for dilution. Adjust the concentration
153	of the	amorphous Teflon solution to 3% (v/v) using the solvent.
154	2.2	
155	3.2	Set the device at a dip coater (Figure 3).
156 157 158 159		Dip coating was adopted here since the uniform coating is indispensable. Rough coating as coating that is too thick can cause deterioration of the atomization efficiency due to SAW ation.
160 161	3.3	Pull down the device so that atomization area is immersed in the solution at a speed of
162		n/s. Keep the device in solution for 15 s.
163	0.2 1111	173. Reep the device in solution for 13 3.
164	3.4	Pull up the device at the speed of 1.7 mm/s. Keep the device in the air for 5 min.
165	J. -	Tall up the device at the speed of 1.7 milly 3. Reep the device in the air for 3 mill.
166	3.5	Pull down the device so that atomization area is immersed in the solution at a speed of
167		n/s. Keep the device in solution for 15 s.
168	012 1111	ing street are device in solution for 15 s.
169	3.6	Pull up the device at the speed of 1.7 mm/s. Keep the device in the air for 30 min.
170	<u>5.5</u>	That up the device at the speed of 117 miny of neep the device in the differ of mini
171	3.7	Bake the device at 180 °C for 60 min using a hot plate.
172		The second of the second of the place.
173	Note:	Thickness of the coating was approximately 400 nm according to quartz crystal
174		palance (QCM) measurement.

4. Experimental Set-up for Atomization

4.1 Mount the SAW device on a printed circuit board.

Note: Since the thickness of the SAW device is 0.5 mm, it can be easily broken. Thus, it is necessary to support it mechanically.

4.2 Measure the frequency characteristic of the SAW device impedance using a network analyzer. The SAW device is connected to the analyzer via a coaxial cable connection of the printed circuit board. Frequency characteristics should show areas where imaginary parts of the device admittance go to zero, which are the SAW device oscillation frequencies.

Note: Check the RF attenuation at the SAW device. The loss of the SAW device increases when it is inappropriately coated. This increase in loss typically occurs due to coating inhomogeneity or excessive thickness of coating; thus, the impedance characteristics before and after coating should be compared. The atomization cannot be performed if the SAW attenuation is too much.

4.3 Connect the SAW device to a function generator through an RF power amplifier.

4.4 Set the waveform of the RF burst signal at a function generator (**Figure 4a**). The burst signal for the SAW device should be a sine wave, and its duty cycle should be 10%. The wave frequency should also be set to the SAW device oscillation frequency obtained from the impedance characteristic measurements.

4.5 Connect a burst square wave generator to a solenoid valve [i.e., micro-dispenser through a driving circuit (Figure 5)] so that a 24 V pulse signal can be supplied to the dispenser, which is also mentioned in the discussion 18,19.

Tip: For driving solenoid valves, a transistor array is convenient. Up to eight solenoid valves can be driven using the transistor array in this study.

4.6 Set the micro-pump to apply pressure to flow the liquid to the micro-dispenser (**Figure 5**). The micro-pump supports the self-priming capability of the micro-dispenser²⁰.

4.7 Use an infrared thermometer to measure the temperature of the SAW device, if necessary.

Note: The temperature at the SAW device surface typically reaches around 45 °C when the RF burst signal (85 Vp-p and 10% duty cycle) is applied for 5 min.

5. Atomization

5.1 Put the liquid (i.e., fragrance or chemical diluted with ethanol) into a vial.

5.2 Set the waveform of the pulse signal applied to a micro-dispenser (**Figure 4b**). The pulse signal is a square wave pulse sequence with a 10% duty cycle and is generated with a function

generator. 221

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Apply the pulse signal to the micro-dispenser to jet a liquid droplet to the SAW device¹⁸. Since a single droplet from the micro-dispenser is only a few nanoliters, a pulse sequence is needed to form a larger droplet for atomization.

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Apply the RF burst signal to the SAW device to atomize liquid droplets¹⁷. The burst signal is applied from a function generator through an amplifier after formation of the liquid droplets. The signal should be applied for as long as vapor is still generated from the atomization process.

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Note: The RF burst signal is used to adjust the average RF power. The SAW device may have a crack if the RF power is much larger than 2 W.

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> 5.5 Observe the surface of SAW device to inspect the remaining liquid droplet.

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Perform the same procedure as done in steps 4.1-4.7 and 5.1-5.5 for a bare SAW device. 5.6 Then, compare the amount of remaining liquid droplet on the coated substrate with that from bare one.

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6. Detecting Scents

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242 6.1 Put the liquid into a vial as done in step 5.1.

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244 6.2 Adjust the height of the SAW atomizer using a jack so that its height remains equal to the 245 participant's nose.

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247 Dispense the liquid onto the SAW device. 6.3

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249 Turn on the fan. 6.4

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6.5 Allow the participant to detect the scent.

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Note: The authors did the sensory test instead of VOC analyzer used in the previous work since the perceived intensity rather than vapor concentration should be evaluated.

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REPRESENTATIVE RESULTS:

257 One microliter of ethanol was placed on both bare and coated LiNbO₃ substrates (ethanol was 258 typically used as a solvent for perfume). A thin film of ethanol solution was formed after it spread 259 onto the substrate (Figure 6a); on the other hand, the sphere-like shape was kept on the coated 260 substrate (Figure 6b). The contact angle of the one microliter of water increased from 50 to 110 261 degrees after amorphous Teflon coating (Figures 6c and 6d). It was found that the amorphous 262 Teflon coating enhanced the hydrophobic nature. The sphere-like shape of the droplet was kept on the coated substrate, whereas the liquid spread into a thin film on the bare substrate.

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Next, the experiment with atomization of 200 nL of lavender was performed (Figure 7). The subsequent images of atomization without and with coating are shown in Figures 7a and 7b, respectively. Timescale in the photos was obtained from the number of frames recorded by a digital camera. Lavender was diluted with ethanol (dilution ratio: 50:1 v/v). On the bare substrate, the liquid spread immediately after it was dispensed. At 33 ms, strong atomization occurred at the center of liquid, whereas only limited mist was generated at the liquid edge within the circle as shown in Figure 7a. At 100 ms, atomization stopped; so, although atomization occurred at first, it stopped soon after. Then, part of the liquid remained. While the solvent evaporated rapidly, partial solute remained on the substrate surface; thus, the remaining solute caused the serious smell persistence. On the other hand, the sphere-like shape with its contact angle of more than 90 degrees was maintained on the coated substrate after it was dispensed (Figure 7b). A concentrated mist was generated during atomization. After atomization, much less liquid in a smaller area was left in the smaller area compared to the bare substrate. Since the remaining liquid was not a smooth and complete surface but instead formed small single droplets, it is difficult to accurately calculate the coverage of droplets on Teflon coating. Roughly speaking, the remaining liquid on the hydrophobic surface was at most 10% of that on the hydrophilic surface.

FIGURE AND TABLE LEGENDS:

- **Figure 1: SAW atomizer.** (a) Configuration of the SAW device and (b) principle of the SAW atomizer. Its electrode consists of gold and chromium. **Figure 1a** is reprinted with permission²⁰.
- Figure 2: SAW device on a printed circuit board.
- Figure 3: Dip coater used in this study.
- **Figure 4: Time sequences.** (a) Waveform of the RF burst signal. The typical values of V_{pp} and T_r are 85 V_{p-p} and 1 s, respectively. A typical duty cycle such as T_H/T_r is 10%. (b) Waveform applied to a micro-dispenser. The typical Tw, T, and N are 1 ms, 10 ms, and 70 pulses, respectively.
- Figure 5: Experimental setup for atomizing liquid droplet.
- **Figure 6: Comparison of liquid droplet shape between bare and coated subtrates.** Shown are a (a) top view of the thin film on the bare lithium niobate surface and (b) side view of the liquid droplet on the coated one. One microliter of ethanol was used in both (a) and (b). Here, (c) and (d) show side views of one microliter of water on the bare and coated substrates, respectively. This figure was reproduced with permission¹⁷.
- **Figure 7: Atomization of the liquid droplet.** Shown are the (a) hydrophilic surface (bare lithium niobate) and (b) hydrophobic surface (substrate coated with amorphous Teflon). The sample is 200 nL of lavender. This figure was reproduced with permission¹⁷.
- **Figure 8: Micro-dispenser based upon a solenoid valve**. Shown are (a) the micro-dispenser's principle and (b) a driver circuit for a single channel. This figure is reprinted with permission²⁰.

Figure 9: Application of the SAW atomizer coated with amorphous Teflon film. Research demonstration from the Intelligent User Interfaces (IUI) Symposium 2018 in Tokyo, Japan.

DISCUSSION:

 One of the key components in this study is the micro-dispenser made of a high-speed solenoid valve^{18,19}. **Figure 8a** shows the principle of this micro-dispenser. The plunger was driven by an electromagnetic coil. Its outlet is completely closed by the plunger during the OFF phase. The plunger rapidly moves to draw liquid in front during a short ON phase, then it moves back to the original location and jets a tiny liquid droplet from an orifice of the solenoid valve, which is driven by the circuit shown in **Figure 8b**. The amount of one liquid droplet is a few nanoliters. The frequency of the valve is between 1 and 1000 Hz, its minimum pulse width is 0.5 ms, and it works much faster than a typical solenoid valve. The typical distance between the orifice of the solenoid valve and the substrate was 15 mm. This study showed that the amount of liquid is precise and reproducible; moreover, it is robust against bubbles.

Smell persistence can be drastically reduced owing to amorphous Teflon coating when an olfactory display based upon a SAW atomizer is used²¹. It can be further improved when a channel dedicated to deliver solvents to the substrate surface for cleaning is used.

The critical step in protocol is manually adjusting the excitation frequency of the atomizer when it deviates from the optimal one. This should be automatically performed in the future. A modification from the initial protocol was to include the silanization process since Teflon coating itself without silanization was atomized.

There are two remaining issues which limit this technique, one being the standing wave problem. The standing wave is generated when reflection occurs at the edge of the substrate. Since antinode and node appears periodically, atomization becomes weak at the node. Although we use a silicone gel to suppress the standing wave, this is not sufficient. A better material to absorb the acoustic energy is necessary.

The second limitation is the durability of the Teflon coating. The Teflon coating is partially removed after atomizing a liquid many times. Since the current condition for coating has not been studied extensively, the authors can optimize it to extend the durability of Teflon coating.

Nevertheless, the significance of the protocol with respect to existing methods is the reduction of remaining liquid after atomization on a surface with coating compared to without coating. Thus, the smell persistence is drastically reduced more than is described elsewhere¹⁷. Using this SAW device, demonstration of the olfactory display was performed. The eight-component olfactory display to demonstrate cassis, orange, whiskey, and their mixture were presented to a user with Head Mount Display (**Figure 9**)¹⁹. In this situation, a SAW device with the proposed coating works well to suppress smell persistence, which otherwise can considerably deteriorate the quality of scent presentation.

The technique described here is important for olfactory display. Moreover, the SAW atomizer is

applicable to a nebulizer for medical use and electrospray ionization for mass spectrometry. The atomization efficiency is also required in those applications.

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DISCLOSURES:

360 The authors have nothing to disclose.

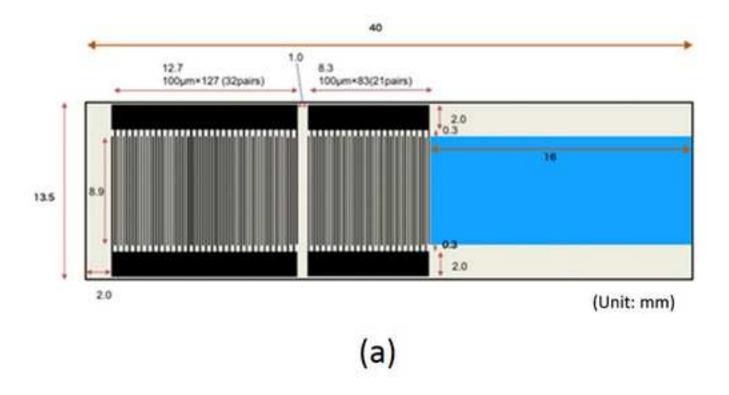
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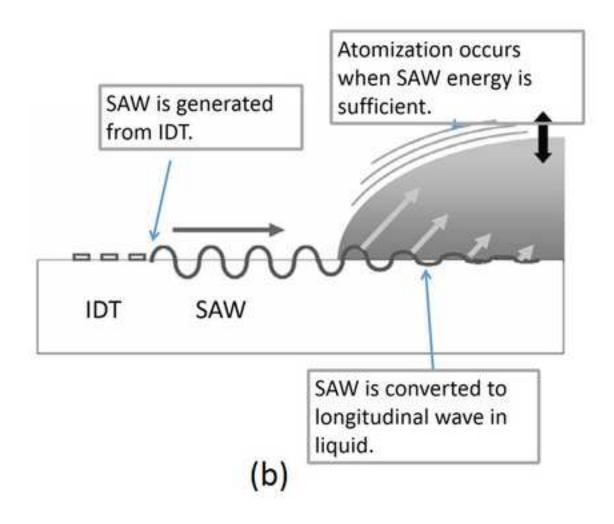
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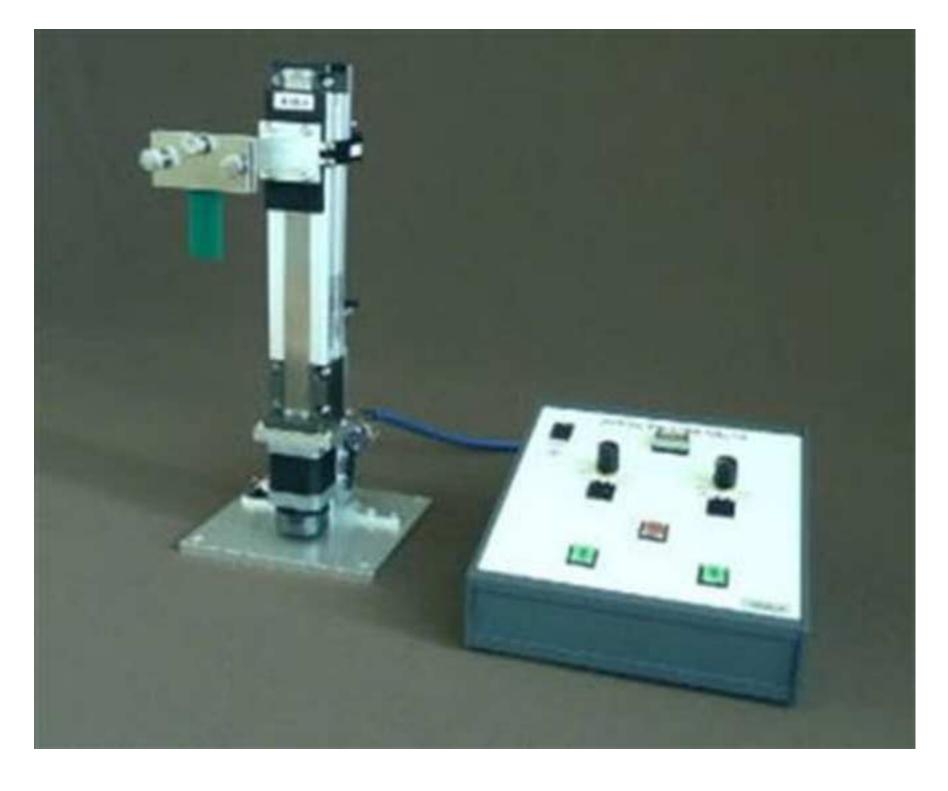
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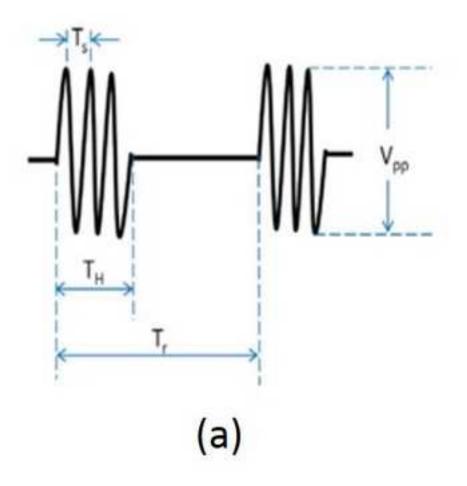
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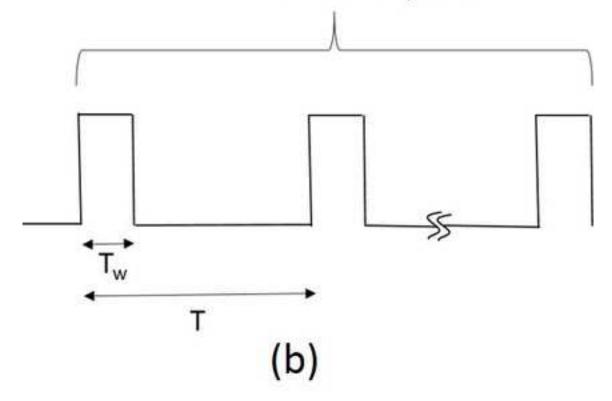


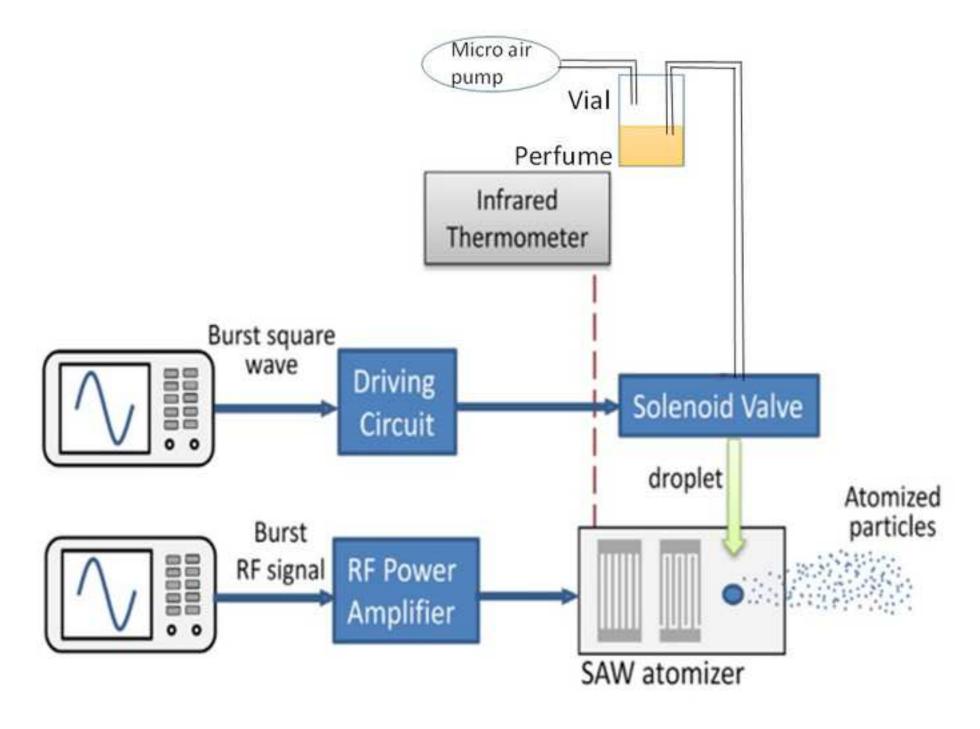


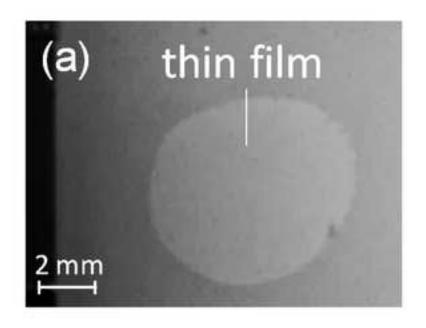


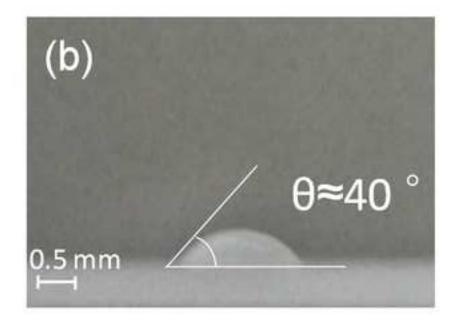


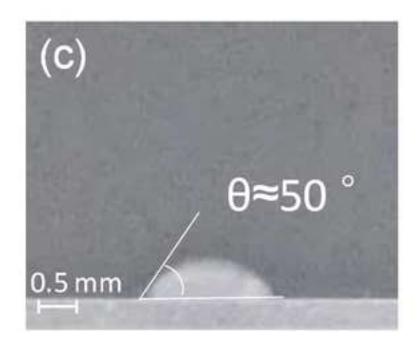
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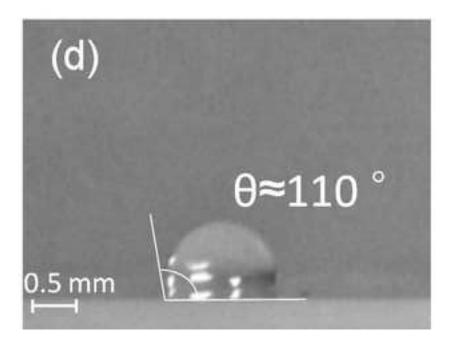


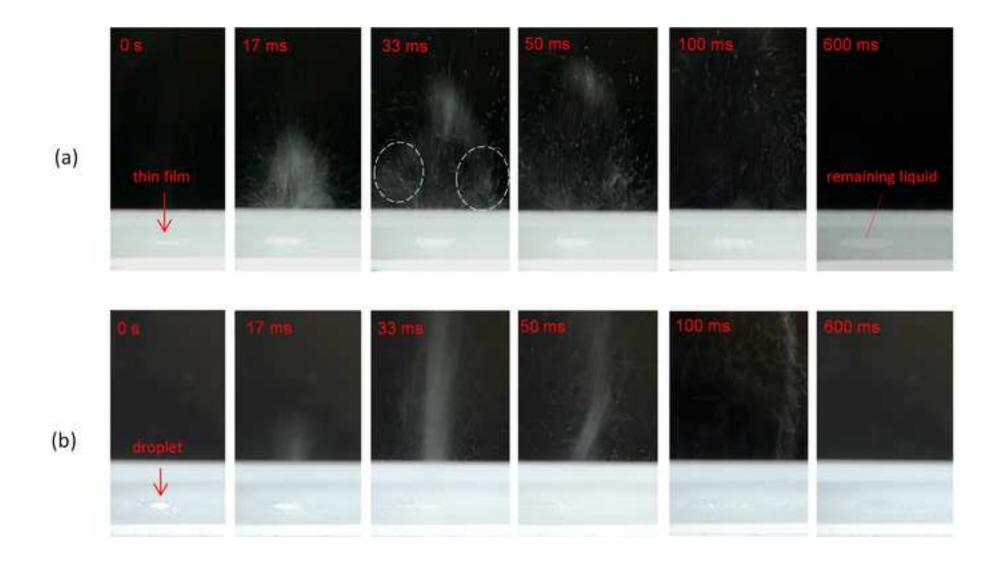


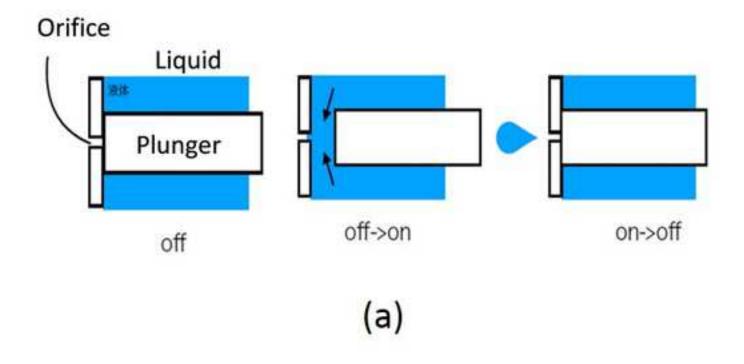


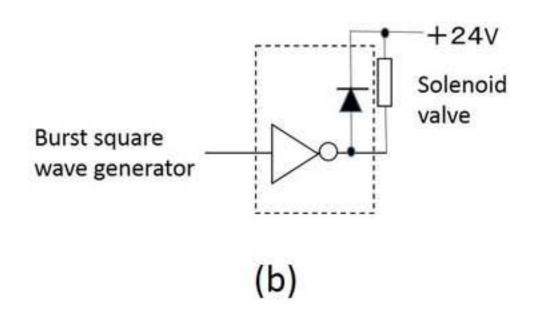














Name of Material/ Equipment	Company	Catalog Number	Comments/Description
SAW device	Lightom		Custom-made
Network analyzer	SDR-kits	DG84AQ VNWA 3E	
Dip coater	Aiden Shin-etsu	DC4300	
Silane coupling agent	Chemical	KBE 903	
Cytop amorphous teflon coating	Asahi glass	CT107MK	
Solvent for diluting cytop coating	Asahi glass	CT-SOLV100K	
Solenoid valve	Lee -	INKA2438510H	
	Texas Instrumen		
Transistor array	t	ULN2803A	
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We checked the manuscript again and revised several points.

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We included doi and arranged the reference format according to your suggestion.

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We highlighted a portion of filmable content in a protocol according to your comment.

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We used standard SI unit according to your comments.

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We include the ethics statement according to your suggestion.

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We revised figures according to your comments.

7. Others

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