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# Nebulization using ZnO/Si surface acoustic wave devices with focused interdigitated transducers

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**Abstract:** Propagation of surface acoustic waves (SAWs) on bulk piezoelectric substrates such as $LiNbO_3$ and quartz, exhibits an in-plane anisotropic effect due to their crystal cut orientations. Thin film SAW devices, such as those based on ZnO or AlN, offer potential advantages, including isotropic wave velocities in all in-plane directions, higher power handling capability, and potentially lower failure rates. This paper reports experimental and simulation results of nebulization behaviour for water droplets using ZnO/Si surface acoustic wave devices with focused interdigital transducers (IDTs). Post-deposition annealing of the films at various temperatures were applied to improve the quality of the sputtering-deposited ZnO films, and 500 °C was found to be the optimal annealing temperature. Thin film ZnO/Si focused SAW devices were fabricated using the IDT designs with arc angles ranging from 30° to 90°.

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Nebulization was significantly enhanced with increasing the arc angles of the IDTs, e.g., increased nebulization rate, reduced critical powers required to initialise nebulization, and concentration of the nebulised plume into a narrower size of spray. Effects of applied RF power and droplet size have been systematically studied, and increased RF power and reduced droplet size significantly enhanced the nebulization phenomena.

**Keywords:** Annealing, ZnO film, Focused IDTs, focused SAWs, nebulization, temperature

## 1. Introduction

Surface Acoustic Wave (SAW) technologies have found many applications in biochemical sensing and detection, drug development, life science and medical research [1-6]. Microfluidic functions such as particle manipulation, concentration, acoustic streaming, mixing, pumping, jetting and nebulization have been achieved by application of radio-frequency (RF) signals to interdigitated transducers (IDTs) patterned on piezoelectric substrates such as quartz, $LiNbO_3$ and $LiTaO_3$ [7-11]. SAW nebulization (SAWN), alternatively referred to as “atomization”, has been studied extensively due to its wide potential applications ranging from targeted drug delivery and inhalation therapy, patterning nanoparticles, deposition of biochemical films, cell product and protein dispersion to mass spectrometry, ionization source for biomolecules [12-17].

Bulk piezoelectric substrates have historically been the SAW substrates of choice for SAWN applications [18-22]. This is mainly due to their good intrinsic material properties such as large piezoelectric coefficient and electromechanical coupling coefficients that result in high energy transduction efficiency. However, they cannot be easily integrated with electronics for embedded control and signal processing, and are not particularly suitable for the development of disposable, flexible or one-time use devices, if compared with thin film counterparts. Bulk piezoelectric materials are generally brittle and prone to crack propagation. This is commonly observed during fabrication processes but also during experiments and operations. Piezoelectric thin-film acoustic wave technology, based on materials such as ZnO and AlN [1, 23, 24], is considered to be one of the key future directions for acoustic wave based microfluidics and lab-on-a-chip devices. With thin-film integration with many substrates including silicon, glass, metal plates/foils and polymer plastics reported, great designs and application flexibility are possible [25-27]. Recently, significant jetting and nebulization were reported using SAW devices made of sputtered ZnO films on silicon substrates [28-31].

Circular, curved or concentric IDTs have been reported to make focused SAW (FSAW) devices [32-34]. Unlike the conventional straight line IDT structures that produce acoustic waves moving perpendicularly to the IDTs, the FSAW devices focus the acoustic wave energy into a small area or a focused point. This could have advantages such as formation of constructive wave interference and maximised mechanical displacements in the focused node between the two focused IDTs. The resulting concentration effects can be harnessed to enhance microfluidic functions, such as jetting or nebulization [32-34]. Propagation of SAW on bulk piezoelectric substrates such as $LiNbO_3$ and quartz, however, exhibits anisotropic effect due to their anisotropic crystal orientations. Thin film SAW devices, such as those based on ZnO or AlN, offer potential advantages, including isotropic wave velocities in all in-plane directions, higher power handling capability, and potential for lower failure rates.

In this work, for the first time, ZnO-based FSAW devices with different curved IDT structures were designed for nebulization and characterised. Post-deposition annealing of sputtered ZnO films were conducted to improve the film quality for the best performance of the

SAW devices. Effects of arc central angle, RF power and droplet size on the nebulization performance were investigated, both experimentally and theoretically.

## 2. Experimental

ZnO films of ~4.5 μm thickness were deposited onto 4-inch (100)-oriented Si substrates using a DC reactive magnetron sputtering process in an ultra-high vacuum system. The base pressure of the chamber was $1\times10^{-4}$ Pa before deposition. A zinc target with a purity of 99.999% was used for deposition of the ZnO films with a 70 mm distance from the substrate. The surface of the substrate was pre-cleaned by means of a short bombardment (5 min) with $Ar^+$ ions using a DC Power of 300 W. The deposition conditions were as follows: DC sputtering power of 400 W; $O_2$/Ar mixing flow ratio of 50/50 sccm; deposition rate of ~750 $\text{nm}\cdot\text{hour}^{-1}$ with a deposition pressure of 1 Pa. Post-deposition annealing was carried out for ten minutes in a nitrogen ($N_2$) gas environment at temperatures of 300, 400, 500, 600, and 700 °C, respectively. Crystal orientations of the ZnO films after annealed at various temperatures were analyzed using X-ray diffraction (XRD, D5000, Siemens) with Cu-$K_\alpha$ radiation ($\lambda$ = 1.5406 Å). Atomic force microscopy (AFM) was used to examine surface morphology and surface roughness of the films, and the microstructure of ZnO films was examined using a scanning electron microscope (SEM).

Curved IDTs patterned using 150 nm thick Al layer were fabricated on the ZnO films (after 500 °C annealing) using a conventional photolithography and lift-off process. Each IDT has 10 pairs of fingers, arranged as concentric circular arcs, with wavelengths of 200 μm and curved angles (or arc central angles) of 30, 60 and $90^o$ as shown in Fig. 1. The IDT reflectors consist of 5 pairs of fingers with same width and gap between the IDT fingers. The distance between reflectors and the IDT is 3/8 wavelength (150 μm for 400 μm and 75 μm for 200 μm wavelength respectively). The resonant characteristics (frequency and S-parameters) of the SAW devices were measured using an RF vector network analyzer (Agilent Technologies, E5061B).

The ZnO/Si SAW devices were placed on a bulk aluminum alloy test-holder during the nebulization experiments to minimize the potential heating effects. RF input signals were generated using a signal generator (Marconi 2024) and then amplified using an RF amplifier (75A250, Amplifier Research) before being transferred simultaneously to the two opposite IDTs of the ZnO SAW devices. The output RF power applied to device was measured using an RF power meter. De-ionized sessile water droplets with volumes of sub-microliters to microliters were placed in the center between the two opposite curved IDTs, and nebulization behavior was recorded using a high speed video camera (VISION Research Phantom MIRO 4) with a capture rate of 4000 frames per second as shown in Fig. 1. The surface temperature of the SAW device after applying the RF power was measured with and without water droplets on the device surface using an infrared video camera (ThermaCAM$^{TM}$ SC640, with a spatial resolution of 0.65 mrad).

## 3. Results and discussion

### 3.1 Optimization of ZnO film Characteristics

To achieve the best nebulization performance, the ZnO film of SAW devices need to be optimized to possess the best material quality, hence the post-deposition annealing at temperatures from 300~700 °C were carried out for ten minutes for the as-deposited films. The XRD spectra of the ZnO films under various annealing temperatures are shown in Fig. 2(a), and the enlarged sections of the diffraction peaks are shown in Fig. 2(b). All the ZnO films show a single peak at a $2\theta$ angle of ~34°, corresponding to the ZnO (0002) crystal orientation, which is needed for good piezoelectric effect of SAW devices. With the annealing temperatures increased from room temperature to 500 °C, the XRD intensity of the film increases, indicating improved crystalline quality and crystallinity of the film. This is because annealing leads to recovery and re-crystallization process where the strained crystallites become relaxed, the number of defects are reduced and the grain sizes are increased [35]. However, when the annealing temperature is over 500 °C, the peak intensity decreases sharply, which is linked with the significant generation of porosity in the film [36].

The stress values of the as-deposited and annealed films were calculated based on a biaxial strain model. The strain $\varepsilon = (c - c_0)/c_0$ along the c-axis, i.e., perpendicular to the substrate surface, was calculated using XRD data (where c and $c_0$ are the lattice parameters of the film and the strain-free lattice parameter of the bulk ZnO, respectively). The obtained values of the strain for as-deposited and annealed ZnO thin films are listed in Table S1 in the Supplementary Information (SI). In order to understand the effect of the annealing on the stress of the ZnO thin films, the film stress values were calculated using the formula $\sigma = -233\times10^{9}\,(c - c_0)/c_0$ [37], and the results are summarized in Table S1. The stress values for the as-deposited ZnO films and those annealed up to 400 ◦C are negative (i.e. compressive stress), whereas with further increase in the annealing temperatures, the stress values are changed from negative to positive (i.e. tensile stress), which is consistent with those reported in literature [35, 37].

With the annealing temperature increases from room temperature to 400 °C, the compressive stress in the films is partially relieved owning to different reasons, e.g., re-crystallization process where the strained crystallites become relaxed; the defects are reduced [36]; and the trapped oxygen atoms are out-diffused caused by thermal expansion [38]. Whereas when the annealing temperature is 500 °C, the film stress value is changed to tensile, and increases continuously with annealing temperature. These could be explained by densifications of the films, increase of stacking faults, and the differences in the thermal expansion coefficients of ZnO and Si [38].

AFM was used to examine the surface morphology and surface roughness of the films over a cross-sectional area of 4 $\mu m^2$, and the results are shown in Fig. 3. The obtained root mean square (RMS) surface roughness values for these films are less than one 7 nm, indicating that all the films are quite smooth, which is beneficial to the good performance of the SAW devices. It was also observed that annealing had little effect on the surface roughness, which is consistent with the previously reported results [35]. A possible reason that RMS changes little may be that the annealing does not significantly affect the columnar structure of the films, and it causes in the decrease of film defects and densification of the films.

To achieve high performance of the SAW devices, the ZnO film should possess good 0002 orientation [39-41]. As the ZnO film annealed at 500 ºC has the largest 0002 peak and a low value of full width at half maximum (FWHM), and at the same time has a relatively lower stress value, therefore, we chose the ZnO annealed at 500 ºC as the testing substrate. The cross-sectional SEM image of the ZnO film after annealed at 500 °C is shown in Fig. 4. It clearly shows that ZnO film has a columnar structure of ZnO nanocrystals, perpendicular to the substrate.

### 3.2 Effects of droplet size and power on nebulization

Using the curved IDT ZnO/Si SAW devices, significant nebulization can be achieved. Fig. 5 shows a sequence of high speed images of nebulization behaviour of a 0.2 µl water droplet driven by an RF power of ~ 51 W at a resonance (or working) frequency of 23.3 MHz using the FSAW device with an arc central angle of 90°. From Fig. 5, three stages of nebulization process can be clearly identified, which are similar to those reported in Ref. [28], but the nebulization is more localized than that using the straight IDTs:

(a) Generation of capillary waves (Fig. 5b). As the leaky SAW propagates into the droplet, acoustic wave pressure drives the liquid droplet, and the surface of the droplet vibrates significantly along with the wave, forming capillary waves on the parent droplet surface;

(b) Generation of ejected droplets (Figs. 5c to 5e). Amplitudes of the capillary waves increase significantly, and the capillary wave quickly becomes unstable. The large amplitude capillary undulations on the free surface of the parent droplet result in whipping, pinch-off and jetting of satellite droplets of about tens to hundreds of microns in diameter.

(c) Significant nebulization with formation of mist and satellite droplets before the consumption of the whole parent droplet (Figs. 5f to 5j). Along with the significant formation and rising of the mist, many large satellite micro-droplets with estimated sizes of tens of microns are generated from the parent droplet.

Fig. 6 shows the effect of droplet size on the nebulization effect with a fixed input RF power of 51 W and a working frequency of 23.3 MHz, using the FSAW devices (with two opposite curved IDTs and a central angle of 90°). When the duration was 2 s, the nebulization was not observed for the droplet with sizes of 0.8 to 1 μL, although significant capillary waves were observed on these droplet surfaces. However, the droplets with the size of 0.5 and 0.2 μL showed significant nebulization. The smaller the droplet size was (for example, droplet of 0.2 μL), the stronger the nebulization phenomena were observed.

The capillary wave theory [42] describes that droplet formation during nebulization is a product of capillary waves on the surface of the excited liquid. When the amplitude of the applied energy is sufficiently large, the crests of the capillary waves break off and tiny satellite droplets are formed. According to Mercer's study [43], the threshold amplitude, $A$, required for the generation of capillary waves is given by:

$$A = \frac{4\mu}{f\lambda} \tag{2}$$

where $\mu$ is the kinematic viscosity of liquid, $f$ is the frequency of acoustic wave, and $\lambda$ is the capillary wavelength. Droplets are formed from the crests of the waves when the amplitude exceeds the threshold amplitude by a factor of about four[38]. The wavelength of the capillary waves induced by a periodic vertical force can be described using a modified form of Kelvin's equation [44]:

$$\lambda = (\frac{2\pi\gamma}{\rho {f_c}^2})^{\frac{1}{3}} \tag{3}$$

where $\gamma$ and $\rho$ are the interfacial tension and density of the fluid, respectively, and $f_c$ is the frequency of the free surface capillary waves. For a water droplet of height of $H$ and lateral dimension $L$, the frequency $f_c$ is given by [44],

$$f_c \sim \left(\frac{H}{L}\right)^2 \tag{4}$$

Before nebulization, for a given droplet, it is roughly $L \approx 2H$ in our situation, which means $H/L \approx 1/2$ for the droplet. However, as the nebulization starts, the value of $H/L$ decreases as the droplet spreads across the substrate surface. It should be noted that a smaller droplet has a smaller $H/L$ ratio compared with a larger droplet thus leading to the smaller value of $f_c$ for the smaller droplet. For example, as shown in Fig. 6, with the droplet size increases from 0.2 to 1.0 µL, the H/L ratio at a duration of 2 s is increased from 1/13 to 1/6. The smaller $f_c$ value for the smaller droplet will then result in a larger capillary wavelength $\lambda$ and smaller threshold amplitude $A$. In other words, nebulization is more easily generated for a smaller droplet, which agrees with our experimental results using the FSAW devices.

With an input frequency of 23.3 MHz, during the stage of capillary waves generation (generally within a few milli-seconds), jetting of large satellite droplets was frequently observed before the starting of nebulization, with examples shown in Figs. 6b to 6d. The larger the droplet size, the higher the jetting height. According to Tan et al [9], SAW could serve to destabilize the air-fluid interface of a droplet atop its surface and hence produce a single elongated column of liquid. When the size of the liquid droplet is decreased, capillary force becomes dominant, thus resulting in more significant capillary waves on the surface of the droplet. Nebulization is much easier to generate when the capillary waves are dominant.

To verify this assumption, the SAW input power was increased to 62 W for the FSAW with a working frequency of 23.3 MHz and central angle of 90°. The results are shown in Fig. S1. Only the 0.2 μL droplet did not show any jetting, with all the larger droplets showing significant jetting phenomena at an early stage of nebulization. Initial jetting of large satellite droplets is not beneficial for nebulization, because the acoustic energy which is coupled into the droplet for nebulization has been partially dissipated and consumed during the initial droplet jetting process. From the above results, we can conclude that there is competition between jetting and nebulization at an early stage of the process, which depends on droplet size, surface hydrophobicity and applied power. What is more, comparing Fig. 6 and Fig. S1, there is an increase of power from 51 to 62 W. This increased power induces a significant jetting effect before nebulization due to the increased surface vibration magnitude on the substrate.

The effect of RF power on the nebulization performance was studied with a droplet size of 0.2 μL, a working frequency of 23.3 MHz and a central angle of 90°, and the results are shown in Fig. S2. In Fig. S2a, at $t$ = 0.2 s, nebulization could be observed only when the RF power was increased above 62 W, and then became significant with further increase of applied RF power. The capillary waves on the droplet surface and its subsequent destabilization to form atomized droplets are governed by viscous-capillary dominated interfacial effects under the concentrated acoustic energy [44]. Large inertial forces ($F_i$) due to the acoustic energy concentration are required to overcome the stabilizing capillary forces ($F_c$) in order to sufficiently destabilize the interface so that nebulization can be generated [44]. When $F_i$ is larger than $F_c$, nebulization can be induced [44]. As discussed in the paragraph above, the droplet size affects the capillary force (*F*$_c$). For the same droplet size, the applied power affects the amplitude of *F*$_i$, which is dependent on a critical value of the acoustic capillary number (*C*). The value of *F*$_i$ increases with the increase of *C* value, which is given by [44]:

$$C = \frac{\rho\omega^2 A^2 cos^2\theta_R L}{\epsilon\gamma} \qquad (5)$$

where $\rho$ is the density of the liquid, *A* the vibration amplitude of the SAW, $\theta_R$ the Rayleigh angle, $\omega$ the angular frequency, *L* the lateral dimension, $\varepsilon$ the ratio of height over lateral dimension and $\gamma$ the tension of the fluid. As the input power is increased, the vibration amplitude of the SAW increases, thus leading to the increase in *C* values and the inertial forces (*F*$_i$). These effects will improve the efficiency of the nebulization. The overall effects of input power and droplet size on the nebulization time are summarised in Fig. 7.

### 3.3 Effects of curved angles on Nebulization

Fig. 8 shows effects of the arc central angle of the IDTs on the nebulization performance, with a fixed working frequency of 23.3 MHz, droplet size of 0.2 μL and power of 62 W. With an increase of the central angle, the nebulization becomes significant as shown in Fig. 8(a-c), which is mainly because of the increase of concentrated acoustic wave energy. The intensity of surface vibration increases, leading to a stronger capillary destabilization and hence result in significant interfacial break-up events and nebulization [14]. Fig. 8d summarizes combined effects of input power and droplet size on the nebulization performance for the various curved SAW devices. With decrease of droplet size, or increase of input power or arc central angle, the time required for nebulization decreases, indicating that nebulization phenomena become more significant. However, the nebulization time does not linearly decrease with the increase of central angle, because it is also a function of applied power and droplet size. For example, when the input power was 42 W and droplet size of 0.2 μL, the nebulization durations were 12.1, 7, and 3.64 s for the central angles of 30°, 60° and 90°, respectively. However, the corresponding values were 3.4, 0.8, and 0.595 s for the central angles of 30°, 60° and 90°, respectively, when the input power was 62 W and droplet size of 0.2 µL. This means that using a large input power, increase of the angle will dramatically reduce the time required for nebulization.

The measured $S_{11}$ parameters of the FSAW devices are summarised in Fig. S3. With the central angle of the IDTs increased from 30° to 90°, the signal amplitudes are increased, due to a significant focusing effect, thus leading to improved piezoelectric responses and increased nebulization effects. The resonance frequency of $S_{11}$ of these FSAW devices for the wavelength of 200 µm is about 23.3 MHz, which corresponds to the Rayleigh mode.

We have done three-dimensional (3D) simulation for the Rayleigh mode using different arc central angle of the IDTs to study the effect of arc angle of the IDTs. The results are present in Fig. 9, which shows that with an increase of the central angle, the surface vibration of curved IDT SAW devices indeed was increased and more significant focusing effects were induced, thus leading to improved piezoelectric responses and nebulization efficiency.

### 3.4 Acoustic heating effect for nebulization

It should be noted that in comparison with our earlier work using a standard IDT design [28] with the same working frequency, the focused IDT SAW device appears to have a smaller transmission signal and a lower nebulization rate. The main reason is that the IDTs used in the Ref. 28 consisted of 30 pairs of fingers which generate a stronger transmission signal. In this work, the focused IDTs consist of only 10 pairs of fingers. Another possible reason is that the ZnO films were deposited using different deposition equipment, thus the film crystal quality might be different.

With such a high RF power applied to the curved IDT SAW device, it might be expected that significant acoustic heating effect would occur on the surface of the device. Fig. 10 shows the surface temperature distribution of the SAW devices without adding water droplet, indicating that the temperature increases with the central angle increased from 30° to 90°. Although the wave displacement on the curved IDT SAW surface is only nanometers or sub-nanometers in amplitude (depending on applied power), the high frequency vibration causes heat generation, especially when there are defects, such as porosity, columnar and small grain boundaries, as well as a rough surface nature of the deposited ZnO films.

In order to identify the temperature distribution on the device surface after applying the large RF power, the surface temperature variations at two positions for each sample were measured. The one is located in the centre of the two curved IDTs and another one next to one of the IDTs as marked in Fig. 10, with a fixed duration of 1.2 s, resonance frequency of 23.3 MHz and input power of 62 W, as well as the central angles of IDTs varying from 30° to 90°. The temperatures in the centre of the two curved IDTs are higher than those near the IDTs, which is attributed to the focused wave energy. As shown in Fig. 10, with the increase in central angle of the IDT, the temperature readings for the SAW devices increase, which is mainly attributed to the larger focused areas and the stronger signal amplitude, thus leading to more acoustic confinement and acoustic energy conversion into heat. Another possible factor could be the electrical resistance, that the 90° curved IDT has larger resistance than other angles.

Figs. 11(a) and 11(b) show the temperature measurement results in the center of curved IDT ZnO/Si SAW device, with and without a water droplet, respectively. Fig. 11c summarizes the measured temperature readings as a function of durations up to one minute. The temperature inside the water droplets increases rapidly firstly, then reaches a steady state with a uniform temperature distribution where the difference from edge to center is less than 1 °C, therefore, only the temperature in the middle point of the droplet is chosen for the comparisons. After the droplet was consumed, the temperature distribution is similar to those without any water droplets on the SAW device.

## 4. Conclusions

Post-deposition annealing of the ZnO films at various temperatures were explored to improve the film quality in order to make a good SAW device, and 500 °C was found to be the optimal annealing temperature. The results show that annealing has improved the quality of the films. Using the optimized ZnO films, the ZnO/Si based curved IDT SAW devices were fabricated and significant nebulization were achieved. Efficient nebulization of water droplets has been investigated using curved IDT SAW devices with various central angles for the IDT structures. Increasing the angle from 30° to 90° significantly improved the nebulization performance, owing to the increased focusing effect and SAW amplitudes. Effects of RF power and droplet size have also been investigated to assess volumetric dependencies to nebulization trends. The results showed that the increasing RF power and decreasing droplet volume both increased nebulization rate and achieved a better nebulization performance. Acoustic heating effects were found to be not significant due to adequate heat sinking and short SAW drive durations employed.

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# Figure Caption

Fig. 1. Schematic of the nebulization experiment of F-SAW devices.

Fig. 2. (a) The XRD spectrums of the ZnO films under various annealing temperatures, (b) the partial enlargement of the diffraction peaks.

Fig. 3. The AFM images of the ZnO films over a cross-sectional area of 4 $\mu m^2$ under various annealing temperatures.

Fig. 4. The SEM image of the cross-section of the ZnO film after annealing at 500 °C.

Fig. 5. Nebulization process at different durations, with the droplet size of 0.2 μL, RF input power of 51 W, arc central angle of 90° and working frequency of 23.3 MHz.

Fig. 6. Influence of droplet size on the nebulization at the fixed input RF power of 51 W, arc central angle of 90°and working frequency of 23.3 MHz.

Fig. 7. Effect of Input power and droplet size on the nebulization time with the fixed working frequency of 23.3 MHz and arc central angle of 90°.

Fig. 8. (a & b & c) the nebulization process of at the duration of 166 ms, with the droplet of 0.2 μL, input RF power of 62 W and working frequency of 23.3 MHz, arc central angle of 30°, 60° and 90°, (d) Effects of input power, droplet size and arc central angle on the nebulization performance.

Fig. 9. 3D simulation of particle vibration and surface deformation of the Raleigh mode with different arc central angles: (a) 30°, (b) 60° and (c) 90°.

Fig. 10 surface temperatures on the SAW devices for the different IDT arc central angle: (a)

30°, (b) 60° and (c) 90° with the RF power of 62 W and working frequency of 23.3 MHz, at the time of 1.2 s.

Fig. 11. Thermal temperature measurement for curved IDT SAW device: (a) with water droplet locating on the center of curved IDT SAW device, (b) without water; (c) Measured temperature readings as a function of durations up to one minute.