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Monitoring SAW-actuated microdroplets in view of biological applications

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ABSTRACT

Droplet localization is of primary importance in (Surface Acoustic Wave) SAW-actuated droplet manipulation for the blind-control of digital microfluidics in lab-on-chips (LOC). This issue is addressed by reconsidering hereafter the implementation of our already published SAW echo method. Two echo signals are now recorded: one for the droplet at its initial known position and one for the droplet at its new unknown position after displacement. Subtracting the delays of the 2 echoes leads to an accurate determination of the unknown position provided that the droplet characteristics remain identical for the 2 considered positions. No detailed analysis of the SAW scattering by the droplet is required with this new protocol. For implementing this echo-localization method, we propose a specific LOC architecture along with software monitoring, suitable for biological applications and devoted here to protein analysis by MALDI mass spectrometry.

1. Introduction

During the past years micro-electro-mechanical systems (MEMS) have been largely developed for fluidic actuation, for example to displace a liquid column in a microchannel or droplets on a plane.

For biological applications, lab-on-chips (LOC) working with droplets have often many advantages compared to more conventional systems. They require smaller quantity of reagents, decrease the risk of handling pollution and display shorter reaction times while being easy to automate. Moreover, droplets with a volume ranging in the nl-μl can be considered as mobile microreactors for the enclosed biomaterial.

Here, the application aims more specifically at protein analysis. Such an analysis is generally performed with a mass spectrometer (MS), which is today a reference instrument. Prior to analysis, the sample must undergo different treatments intended for purification, protein digestion into peptides and concentration. Once the preparation is achieved, the peptides are electrically charged in order to enter the spectrometer. Two methods are currently used: ElectroSpray Ionization (ESI) and Matrix Assisted Laser Desorption Ionization (MALDI) where matrix is added to the droplet sample to allow a laser targeting.

Digital microfluidics [1] is thus especially suitable for protein analysis by MALDI-MS. Two methods are convenient for the 2D displacement of liquid droplets: the first one being ElectroWetting On Dielectrics (EWOD) and the second one, Surface Acoustic Waves (SAW).

EWOD necessitates a surface padded with electrodes. A step-by-step displacement of a droplet is triggered by a driving electrical field resulting from the application of a potential of some tens of volts between 2 adjacent electrodes. No droplet localization method is thus necessary. However, droplet contact angle under the applied potential is limited and reaches a saturation value beyond which increasing the voltage remains inefficient to move a pinned droplet.

The SAW method does not show the previous drawbacks. The displacement area is free of electrodes and the actuation force mainly depends on excitation power. To cope with the transport of droplets over pinning surface defects or hydrophilic areas, the additional SAW power reserve can be used. The main constraint is the need of a piezoelectric substrate such as $LiNbO_3$. However such a substrate just like Pyrex® allows surface treatments to be performed in order to ease droplet displacement.

SAW are commonly used in RF signal processing, especially for filters in mobile communications. But SAW can also be used for conveying small objects [2,3]. In such applications, the object moves in the direction opposite to the surface wave propagation. SAW are now also used for microfluidics in many fields [4,5]. Thus atomizators [6–8], systems for particle collection and concentration

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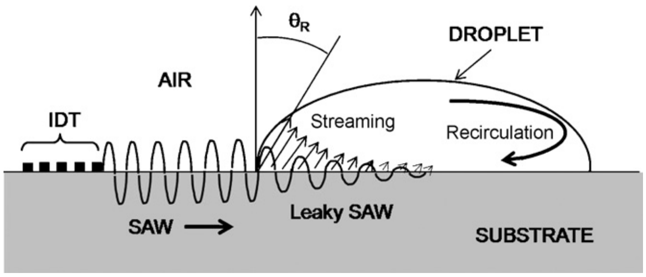

Fig. 1. Schematic illustration of the Rayleigh SAW on the surface of a piezoelectric substrate and its interaction with a liquid droplet. The acoustic energy is radiated into the fluid at an angle θ_R, leading to internal streaming in the small fluidic volume.

[9–12], mixers [13,14], PCR systems [15], heaters [16] and systems for lining carbon nanotubes [17,18] are examples of variant applications.

In digital microfluidics literature [19–24], systems for droplet displacement can also be cited. In such systems, SAWs are simply emitted by interdigital transducers (IDTs) [25]. When hitting a droplet, the major part of the incident wave energy radiates into the droplet along a direction pinpointed by θ_R, the so called Rayleigh angle, while only a leaky wave propagates under it (Fig. 1). The radiated energy induces streaming, which finally results in droplet displacement when the acoustic power is sufficient. It should be noted that this displacement occurs along the SAW propagation direction. During transport, the droplet deformation [26], reflecting the complexity of the SAW energy propagation and scattering, is part of the transport phenomenon itself. In our study, we limit the importance of this deformation by using low-power SAW so that the droplet integrity is preserved. In such conditions droplet displacements can be clearly measured.

We focus here, on the development of a platform for SAW displacement and localization of droplets in view of MALDI-MS protein analysis. The goal is to blind-control the droplet trajectory so that it can contact functionalized micropads laid on the surface along a preset path. This ensures the droplet-enclosed proteins undergo the necessary and specific biological treatments. Therefore droplet localization is crucial.

Few droplet localization methods have been published. Alzuaga et al. [27] have proposed the use of a substrate including many opposite pairs of IDTs distributed around the working area. When a droplet is actuated between a given pair (emitter–receiver), the acoustic signal sent by the emitter is attenuated and this attenuation, detected by the receiver, allows localization along one direction. Wu and Chang [28] used slanted IDTs allowing frequency sweeping. To determine the droplet position, the measured frequency of the absorption dip in the reception signal is determined. Such methods require special technological realizations or frequency sweeping and accordingly additional fabrication or implementation problems.

Our present paper follows two others [25,29]. The first one [25] deals with the controlled 2D actuation of a liquid droplet by SAW. It is shown that the platform built from a $LiNbO_3$ substrate fitted with IDTs allows the controlled transport of droplets, provided that substrate hysteresis is reduced by chemical treatment. The droplet can then move freely on one plane or constrained between two parallel planes. In view of combining SAW actuation and localization, the use of a pulsed actuation (on–off keying) is proposed thus leading to a step-by-step droplet progress instead of a continuous one. Hydrophilic zones are also patterned on the substrate so as to simulate the microfluidic behavior of bio-functionalized micropads present in the LOC. It is pointed out that droplets can be correctly de-trapped when these pads are fitted with hydrophilic microtracks at their outlets. It is concluded that in these conditions, SAW excitation can be a solution to the droplet handling in a LOC devoted to physico-chemical applications. However, making an operational LOC still requires a droplet localization method for the blind-control of the working system.

The second paper [29] deals with a SAW echo-localization method. Only one IDT working as an emitter–receiver is enough for droplet actuation and localization along one direction. Indeed an actuation signal is sent for the step displacement of the droplet then a localization signal is sent and its echo is recorded. We have tried to determine the droplet new position from the echo propagation delay by setting the emitted localization pulse as a time origin. Such a principle necessitates studying the SAW propagation at once both in the substrate and inside the droplet. But whereas SAW propagation in the solid is well known, the situation is more complex inside the droplet where multi-reflection or even resonances [30] occur. A relation based on a coarse model of the situation finally led to determinations of droplet positions generally not enough accurate. To improve the results, we proposed to use a constant corrective quantity. But as long as the study of the echo building and of its time delay is left unaccomplished, adding this constant remains an ad hoc procedure. Moreover, odds are that this correction would have to be adjusted according to the droplet liquid composition. For biologic applications, where precisely this composition alters during the droplet treatment the validity of such a correction becomes questionable.

The goal of the present paper is thus to reconsider the problem of determining droplet position and to show that it is possible to use the echo-localization method without resorting to an ad hoc correction. The new procedure presented here consists of using the duration between two echoes sent by the droplet at two different locations: a known departure location (termed location 1) and an unknown arrival location to be determined (termed location 2). To the extent that the droplet characteristics remain unchanged between these two positions, it is possible to avoid the detailed study of the echo by using the propagation time between 1 and 2, contrary to [29]. It is then shown how to implement this method by proposing a novel LOC architecture to achieve MALDI-MS analysis and a dedicated monitoring bench.

2. 2D Transport of droplets

In our previous article [25], the fabrication of a SAW platform designed for liquid microdroplet transport has been reported. As one IDT allows transport in one direction, a pair of two opposite IDTs is useful for more flexibility. As shown in Fig. 2, the platform is fitted with four pairs of IDTs for 2D displacement of the droplets along two perpendicular axes all over the available area.

An X-cut $LiNbO_3$ piezoelectric substrate is chosen for its good electro-mechanical coefficients along both perpendicular Z (K^2 = 5.9%) and Y (K^2 = 3.1%) directions. These values for K^2 along the two axes are more balanced than those for the typical 128Y-X cut (K^2 = 5.6% along X and K^2 = 1.2% along direction perpendicular to X). An IDT allows both the generation and detection of SAW on the surface of a piezoelectric substrate, thus performing direct conversion of electro-mechanical energy via piezoelectricity. The transducers used in our devices are fabricated using a lift-off process. They have 20 fingers with a pitch of $2d = 180\ \mu m$ (see Fig. 3). To avoid SAW dispersion due to the mechanical loading from surface metallization, the finger thickness (here 0.6 μm, 1000 Å of Ti and 5000 Å of Au) is designed to be much smaller than $2d$. In order to minimize spurious wave reflection, an adhesive gel is attached to the wafer rim, acting as an acoustic absorbent.

The resonance frequency f_0 of an IDT is linked to the wave propagation speed V and the finger period $2d$ by the following relation