Controlled Spalling In Lithium Niobate: A Universal Mechanical Release Technology For Inorganic Thin-Film Devices

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I. Abstract

Scalable and cheap fabrication of thin films (less than 50 μm thick) has the potential to revolutionize aerospace, bioengineering, and consumer electronics with small and flexible electronic devices. Controlled spalling, a method capable of producing these benefits and more, controls the stresses within a substrate and allows for large-scale release of the desired layer. In this work, controlled spalling is implemented in a laboratory setting using electroplating to produce these stresses, then analyzed and refined while applying it to the case of thin-film lithium niobite transducers.

II. Introduction

Thin-film lithium niobate ($LiNbO_3$) structures have been shown to be extremely useful as surface acoustic wave (SAW) transducers [1]. These transducers are useful as mobile communication bandpass devices, and, in particular, transducers operating from 3-30 GHz, the so-called super high frequency (SHF) range, allowing for faster data transfer rates [2] as well as greater sensor selectivity [3]. $LiNbO_3$ thin films have been demonstrated to have high coupling coefficients (K^2) and stability at frequencies in the SHF range due to the material's piezoelectric properties [4]. However, to achieve these performance metrics, conductive interdigital transducers must be fabricated on the $LiNbO_3$ surface. In this work, we concern ourselves with the fabrication of these thin-film transducers. A method of creating thin-film $LiNbO_3$ metal transducer devices easily and cheaply would lead to more accessible technology for a wide variety of communications and MEMS applications.

$LiNbO_3$ single-crystal thin films can be deposited by chemical vapor deposition on other crystalline materials, most commonly silicon [5]. However, production of thin-films in this manner requires additional deposition and etching. Etching of $LiNbO_3$ is a volatile process that can lead to contamination of other samples, and is best avoided. We propose a better approach: to release a thin film from a bulk substrate after fabrication of that device.

In the past several decades, various methods for brittle thin-film release have been explored. Physical release methods such as epitaxal lift-off [6], which selectively etches a substrate from a thin film, limit the variety of materials that can be used and can damage devices if the etchant is not compatible. Optical methods, meanwhile, include release methods such as laser lift-off [7], which aims a tuned laser at a film-substrate interface which is excited to release upon light absorption. Optical methods are expensive and limited in scalability due to the small-area beam shape. I address the need for a universal mechanical release method, opening the door to $LiNbO_3$ thin-film devices at low cost and accurate thickness.

III. Dynamics of Controlled Spalling

Controlled spalling is understood by separating the stress at the crack tip into directional modes, each with a well-defined role [8, 9], as shown in Fig. 1. Mode I stress, the component of the stress perpendicular to the tensile layer, acts to open the crack. Mode II stress, meanwhile, is the shear stress acting directly at the crack tip, parallel to its propagation.

The magnitudes of both stresses, denoted K_I and K_{II}, are calculated according to Eqns. (1) and (2) [8].

$$K_I = \frac{P}{\sqrt{2Uh}}\cos\omega + \frac{M}{\sqrt{2Vh^3}}\sin(\omega+\gamma) \quad (1)$$

$$K_{II} = \frac{P}{\sqrt{2Uh}}\sin\omega - \frac{M}{\sqrt{2Vh^3}}\cos(\omega+\gamma) \quad (2)$$

Suo et. al. [8] finds that cracks deflect in a direction that decreases K_{II} as shown in Fig. 2. The result of any crack propagation, therefore, will be a unique, constant depth of cracking (and thin-film thickness) at which mode II stress is eliminated.

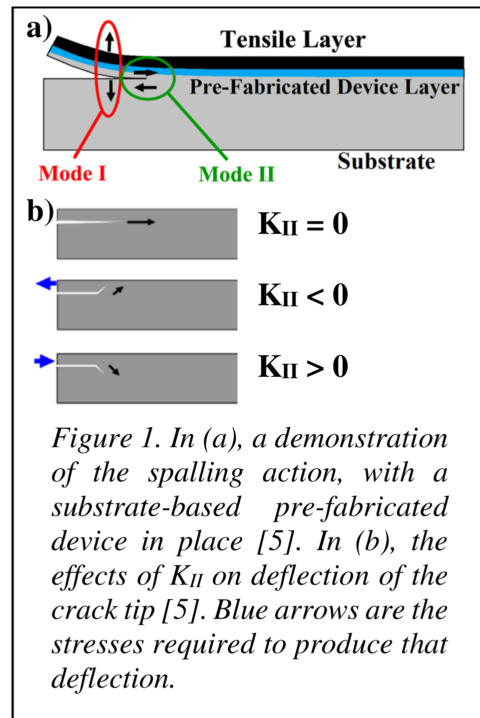

Figure 1. In (a), a demonstration of the spalling action, with a substrate-based pre-fabricated device in place [5]. In (b), the effects of K_{II} on deflection of the crack tip [5]. Blue arrows are the stresses required to produce that deflection.

a. Application to $LiNbO_3$

$LiNbO_3$ is brittle, so this theory applies. If one can find a way to manipulate the stresses in a pre-fabricated $LiNbO_3$ device, a thin film can be created with excellent control of thickness based on the calculation of K_{II} stress. It is additionally convenient in that the required contacts for a SAW transducer can be fabricated on the substrate before creation of the thin film, greatly easing the process.

IV. Overview

When the summer began, I had already designed a methodology for the spalling of silicon wafers, to cheaply test all components of the process before expanding to $LiNbO_3$. The process was not yet consistent, but after the work of this summer that has begun to change. It consists of three major steps: the deposition of the stressed layer so that spalling can be made possible, the characterization of the system to ensure everything will occur as expected, and spalling itself, where the thin film is peeled up using the stresses imparted by the deposited layer. This summer, these steps were tackled systematically and in order so that consistent, predictable results could be achieved.

a. Stressed Layer Deposition

I chose electroplating, the transfer of Ni+ ions from solution to a negatively biased substrate, to meet the significant challenge of controlling the thickness and stress of the final tensile layer. I

carefully designed a 3D-printed, substrate-specific holder to take into account the variable curvature of the sample due to its internal stress. The result allowed for easy mounting and minimal handling while maximizing conductive contact with the sample. However, direct electroplating on Si substrates causes failure almost immediately.

The challenge of adhesion of the tensile layer to the substrate was met in two ways. First, a thin Ni 'seed' layer was deposited with electron beam evaporation, a high-adhesion method in which a gas of the deposition atom contacts the substrate. Second, adhesion was enhanced with a layer of titanium, which bonds with both the substrate and Ni, deposited between the two materials.

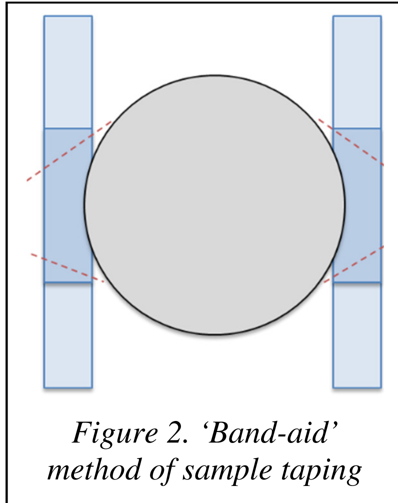

Figure 2. 'Band-aid' method of sample taping

Over the summer, however, I found that the deposition of this seed layer was very inconsistent. I narrowed this down to two problems: first, the deposition system had poor beam control, causing inconsistent layer quality, and second, samples tended to break when removed from the double-sided tape that allowed them to hang upside-down in the deposition chamber. I solved these problems, and rendered deposition consistent, by switching to a new system with better control and developing a method of taping that was easy to remove (shown in Fig. 2). The darker portions, which aren't sticky on the bottom, can be cut along the red lines so that the sample can be lifted from the surface and peeled up. This method is original and applicable to any breakable sample.

I also noticed that, in some cases, the seed layer tended to peel up. I attributed this to cleanliness: particles that get between the layers and interfere with adhesion. After implementing piranha cleaning before deposition, this problem disappeared, leaving seed layer deposition consistent.

Electroplating, then, needed to be refined. Many samples were electroplated, and the problem of electrical contact became apparent. A holder was designed and 3D-printed that uses o-ring seals to prevent nickel deposition on a metal ring that holds the sample down and provides that contact. With this, electroplating area was decreased, but all failures from the edge were eliminated and plating became quite consistent.

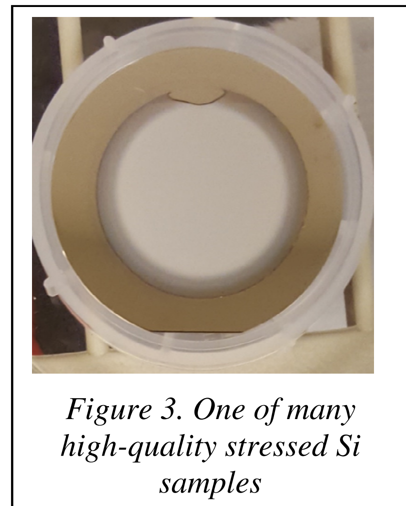

Figure 3. One of many high-quality stressed Si samples

After this, Nomarski microscopy was used to detect defects in the film, and many were found. After testing several theories, it was found that rinsing the samples with water after deposition would prevent the electroplating solution from remaining on the wafer and dissolving the nickel layer. Implementing this rinsing resulted in flawless, consistent samples, allowing me to move on to the analysis step.

b. **Stressed Sample Analysis**

Two methods of sample analysis after plating were developed. The first of these is designed to analyze the sample's thickness uniformity. The nickel is selectively etched away so that a step height from the top of the electroplated layer to the silicon below can be measured using a profilometer at many different places through the center of the wafer. A wafer so etched is shown in Fig. 4a, and the results of this etching at many different times of plating are shown in Fig. 4b.

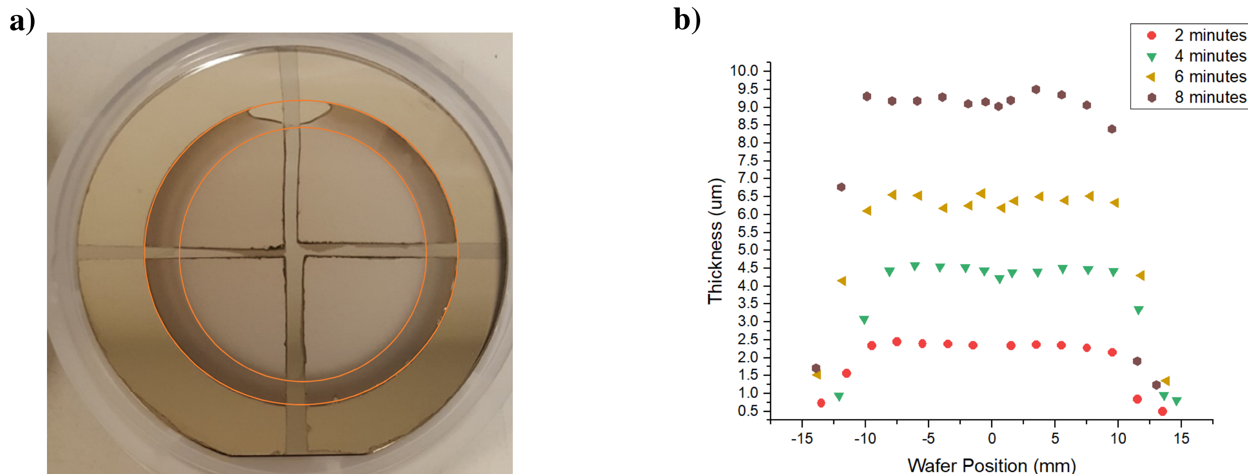

Figure 4. Results of refined etching and thickness uniformity analysis

Results suggest excellent thickness uniformity except around the edges. This 'drop-off' area is highlighted in Fig. 4a, and is almost certainly due to electroplating inhibition by the o-ring. It is yet to be seen if this non-uniformity will affect spalling.

The second method of analysis optically measures the curvature of the wafer surface, then uses Stoney's equation to calculate stress [10]. Although the machine used to perform this measurement is difficult to understand, it was verified through other methods and I believe it is well-understood. The results of stress measurements, as well as averaged thickness measurements from etching, on different plated samples is shown in Fig. 5.

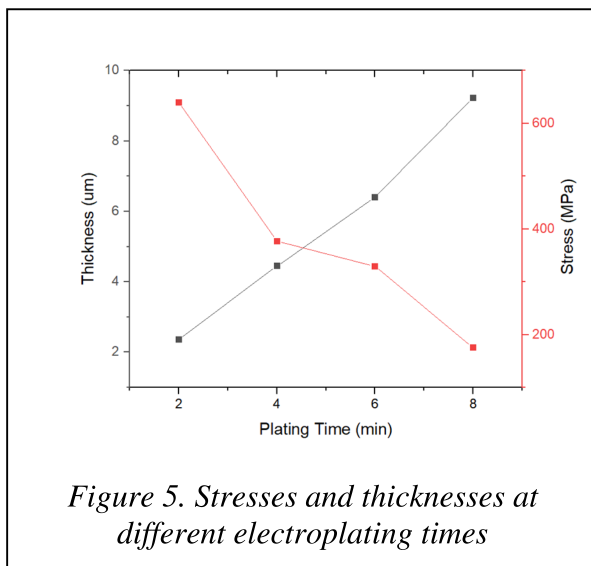

Figure 5. Stresses and thicknesses at different electroplating times

V. Conclusion and Future Work

Although this research is not yet complete, results are very promising. Consistent spalling, along with stress and uniformity results that align well with theory, are indicative of a process that is robust and well-suited to incremental adjustment to achieve controlled spalling in $LiNbO_3$.

Analysis of these results based on theory [8] indicates that spalling will not occur at these levels of stress. To combat this, the solution will be changed in several ways to increase stress. First, I have observed a decrease in acidity over the 171 consecutive minutes of electroplating that have taken place this summer. Since chlorides increase deposit stress [11], it is reasonable to conclude that the chloride concentration in the bath has been taken up by previously free-floating H^+ ions and decreased stress. Stress can also be increased by the addition of ammonia to the bath [12], so this will be investigated as my research continues.

The third step in the process, initiation of controlled spalling, could not be reached this summer, since stress was lower than expected. However, a design for inducing spalling is in place, and will be tested once stresses are brought up. I will continue this research in ECE 499, and I am optimistic about my results.

VI. References

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