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Grinding free electric-field poling of Ti indiffused *z*-cut $LiNbO_3$ wafer with submicron resolution

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ABSTRACT Electric-field domain inversion cannot be performed in *z*-cut $LiNbO_3$ after waveguide fabrication using common Ti-indiffusion techniques. In this work we show that an appropriate combination of low indiffusion temperature, dry O_2 and Li enriched atmosphere during waveguide fabrication allows subsequent domain inversion without the need for any surface grinding, which would dramatically increase the risk of $LiNbO_3$ substrate breakage during its processing. The proposed technique allows a simplified, robust and high yield processing over full wafer scale (up to 4″) with sub-micron resolution.

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1 Introduction

In ferroelectric materials (e.g. $LiNbO_3$), the second-order nonlinear optical properties (including electro-optic and nonlinear optical properties) are intrinsically related to the crystal orientation (also called poling orientation or domain) [1]. In particular $LiNbO_3$ substrates up to 5″ in diameter which are poled during or after crystal growth are available. These wafers present a single domain structure, i.e. the same *c*- or *z*-axis orientation over the entire volume. It is also known that by applying high electric fields (> 20 kV/mm) through appropriate electrodes across single domain $LiNbO_3$ crystals, one can achieve domain inversion [2]. Electric-field domain inversion is widely exploited in, for example, quasi-phase-matched nonlinear optical frequency conversion processes, such as second-harmonic generation [3], optical parametric oscillation [4] and wavelength switching [5]. More recently, domain inversion has been used in integrated electro-optics, both in a periodic [6] and non periodic fashion [7], to produce high frequency narrow band modulation and low driving voltage broadband modulation, respectively.

For both integrated nonlinear frequency conversion and electro-optic devices one needs a suitable means to produce waveguides. The two most common techniques to produce waveguides in $LiNbO_3$ are proton exchange and Ti indiffusion. Both of them are used for nonlinear frequency conversion devices while Ti indiffusion in fact is the preferred one to be used in high frequency (> 1 GHz) electro-optics, mainly thanks to its superior microwave performance. It has been recently shown that the performance of integrated electro-optic modulators in Ti indiffused *z*-cut $LiNbO_3$ can be significantly improved by making use of domain inversion. In particular, modulators with driving voltage well below 3 V [7–9] for inexpensive electro-absorption drivers as well as chirp free modulators [10] can be produced.

In Sect. 2 we report the investigation of the physical parameters affecting the feasibility of electric-field poling just after waveguide fabrication, and we provide a recipe for obtaining a grinding free process. In Sect. 3 we report an example of a device where our grinding free process has been employed. Finally, in Sect. 4 conclusions are drawn.

2 Grinding free domain inversion process

It is known that Ti indiffusion is accompanied by Li_2O outdiffusion from the crystal surface [11]. If ad-hoc measures are not taken to prevent Li_2O outdiffusion, domain inversion on the Z^+ crystal surface occurs: the result is usually a thin (up to a few tens of μm) domain inverted layer, which is oppositely oriented with respect to the initial crystal domain structure. The closer the temperature to the Curie point (1145 °C) the more significant the formation of the thin domain layer on the Z^+ surface [12]. In addition, it is known that the Ti indiffusion process itself can cause domain reversal on the Z^+ [13]. For these reasons Ti indiffused waveguides are produced on the Z^- surface.

So far it has been shown that, after the Ti waveguide is made, the thin domain inverted layer usually prevents domain inversion [14]. This is most likely related to the fact that the thin layer acts as an insulating layer for the poling. Indeed, during poling the internal polarization switching current is compensated by the external ohmic current which has to reach the surfaces of the crystal portion that are to be inverted. The presence of a thin layer which is already oriented as the applied field, and at the same time is insulating and does not allow any current to reach the Z^+ surface of the initial crystal, which thus does not invert. A direct method to solve the problem is grinding of the Z^+ surface to remove the thin domain inverted layer and bring the crystal to a thinner single domain structure with optical waveguides on the

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z-surface [5, 14]. After removal of this thin layer the crystal can be poled. However, beside being time consuming and expensive, the grinding process can be detrimental for the mechanical resistance of the crystal, in particular when most of the processing is performed at a wafer level before dicing into chips. Any crystal weakness introduced by grinding may result in breakage of the wafer during subsequent processing steps with a consequent loss of all the chips.

In this paper we investigate the physical parameters of the Ti indiffusion process and provide a recipe which allows subsequent poling of the full wafer without the need for grinding the crystal surfaces after waveguide fabrication. In particular we show how the settings of Ti indiffusion temperature and atmosphere is critical to avoid the grinding process, which is time consuming and introduces high risks of breakage.

Our belief is that the simultaneous use of low temperature (1030 °C) and O_2 atmosphere avoids the need for any grinding. The introduction of dummy crystals of $LiNbO_3$ in the O_2 enriched atmosphere that act as sources of Li_2O has the effect of reducing the out-diffusion for the fabricated wafers. At the same time a lower temperature (i.e. a temperature farther from the Curie point) reduces local domain inversion at the Z^+ surface (the lower the temperature the higher the required local coercive field to invert the crystal). The final result is an insignificant domain inverted layer on the Z^+ face.

These hypotheses are confirmed by poling measurements carried out on four different samples from the same wafer, as summarized in Table 1: (#1) crystal as received, no Ti indiffusion; (#2) crystal with Ti indiffusion at 1030 °C, 9 h, 1.3 l/min O_2 flow and Li atmosphere; (#3) crystal with Ti indiffusion at 1030 °C, 9 h, 1.3 l/min N_2 flow and Li enriched atmosphere; (#4) crystal with Ti indiffusion at 1100 °C, 9 h, 1.3 l/min O_2 flow and Li enriched atmosphere.

The four samples have a quite different poling behavior (Figs. 1 and 2). Figure 1 shows the poling current dynamics for #1, 2 and 3 subjected to subsequent 10.5 kV, 4 s pulses. It is evident that #1 (as received) and #2 (1030 °C, O_2 flow) show a faster dynamic than #3 (1030 °C, N_2 flow) which is unable to complete poling. In particular #1 also finishes poling before #2 (due to higher poling current) and #3 shows a decreasing poling current (negligible already after 5 pulses). The difference is also highlighted in Fig. 2 where it is evident that #1 completes poling (reaches a total charge of about 190–200 μC) before the end of 4th pulse, #2 before the end of 5th pulse, while for #3 the cumulative charge is saturating at about 50 μC, i.e. 25% of the total charge required to invert the whole area (same as #1 and 2).

Sample #4 (1100 °C, O_2 flow) shows a very low poling current (< 0.5 μA) during the first poling pulse at 10.5 kV (Fig. 1). To see whether poling efficiency could be increased

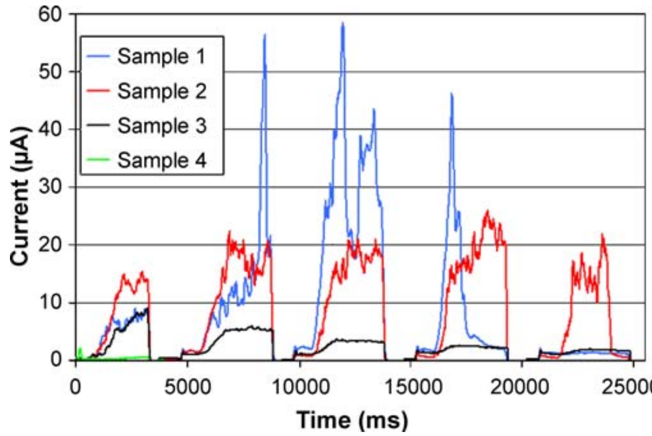

FIGURE 1 Current during poling for samples: #1 (as received), #2 (1030 °C, O_2 flow), #3 (1030 °C, N_2 flow) and #4 (1100 °C, O_2 flow)

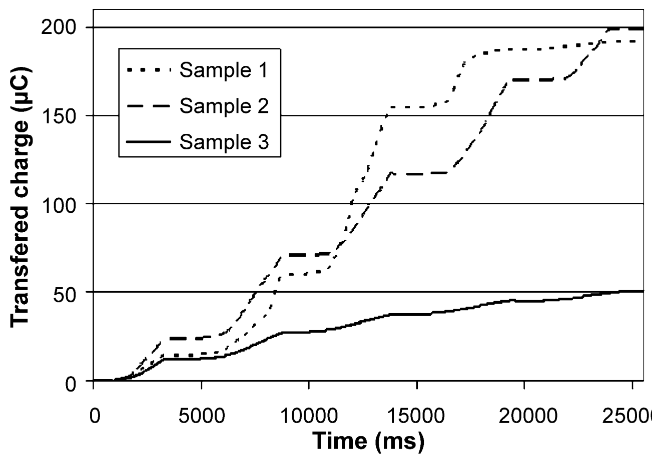

FIGURE 2 Cumulative poling charge for samples 1, 2 and 3

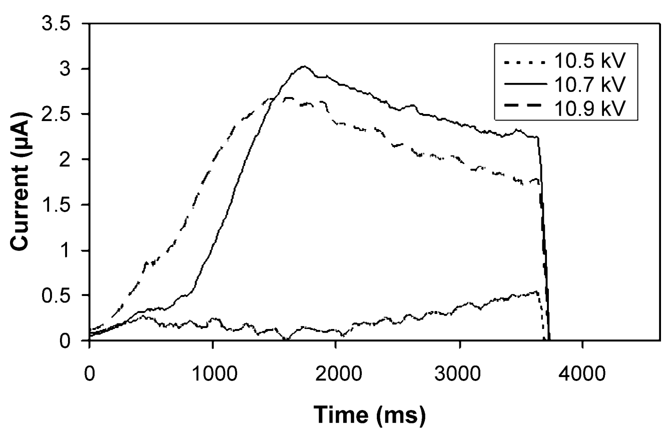

FIGURE 3 Poling current of sample 4 at increasing applied voltages where it evident that the poling current is negligible even for higher voltages (before breakdown)

at higher voltage pulses, subsequent poling was performed at 10.7, 10.8, and 10.9 kV, until breakdown occurred at 11 kV. Even for these higher voltages the poling current was always limited to < 3 μA, showing the impossibility of getting significant poling in this sample (see Fig. 3). Note that, similarly to #4, #2 above was also subjected to higher voltage poling after the 5th pulse at 10.5 kV without getting any significant increase in current (cumulative charge).

Sample #	T (°C)	Atmosphere	Flux (l/min)
1	–	–	–
2	1030 (9 h)	Li, O_2	1.3
3	1030 (9 h)	Li, N_2	1.3
4	1100 (9 h)	Li, O_2	1.3

TABLE 1 Values of temperature and atmosphere composition for the four Ti indiffused samples

Contrary to what has been reported in the literature so far, we achieved a waveguide fabrication process that does not prevent subsequent poling. After deposition of a Ti metal strip on the Z^- face of a 0.5 mm 3″ wafer, Ti indiffusion is performed at 1030 °C for 9 h under an O_2 flow of 1.3 l/m. During the indiffusion dummy $LiNbO_3$ wafers are added to enrich the atmosphere with Li. Without any grinding process the wafer is then patterned with an appropriate insulating masked layer and poled using 10.5 kV pulses. The regions where LiCl water electrodes are in contact with the free crystal surface are domain inverted without any significant difference with respect to wafers as received (not indiffused with Ti).

3 Example of application: electro-optic modulator

As an example of an application of the process described, Fig. 4 shows a typical result of a structure with Ti indiffused waveguides and domain inversion revealed by differential etching. The two optical waveguides of a Mach–Zehnder modulator with a distance of about 8 μm between their inner sides are located in oppositely oriented domain regions, with the domain inverted boundary being clearly in the middle. The uniformity of poling and waveguides over the full 4″ wafer ensures a high yield (currently 95%) for the overall chip fabrication. In fact a detailed analysis over the full wafer has revealed a precision of ±0.7 μm of domain wall positions with respect to the initial mask for the patterning of the insulator. The structure in Fig. 4 has been used to realize a 10 Gb/s integrated electro-optic modulator, single drive and chirp free, with a switching voltage of about 2 V, reachable by inexpensive drivers typically used for electro-absorption devices [9]. We have not seen any difference in the fiber-to-fiber optical loss of our low temperature Ti-indiffusion process with respect to a higher temperature one. In fact, for our devices, we measured a typical fiber-to-fiber optical loss of about 2 dB for straight waveguides and 3 dB for a Mach–Zehnder interferometer.

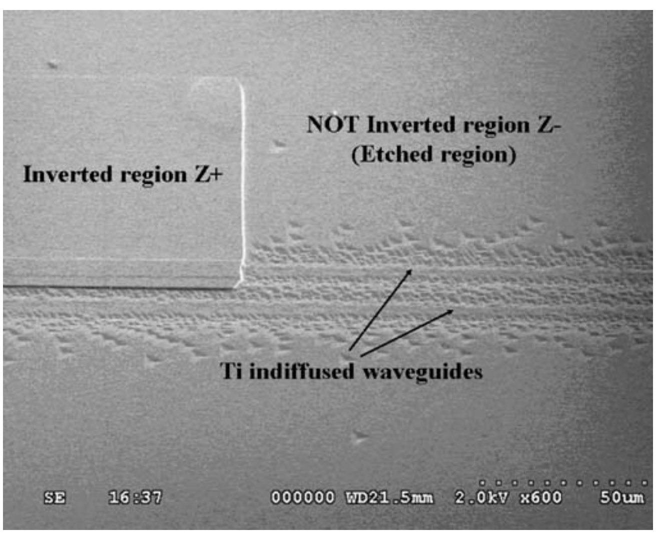

FIGURE 4 Domain inversion in a Ti indiffused crystal revealed by etching: the domain inversion boundary is clearly in the middle of two optical waveguide Mach–Zehnder arms

4 Conclusions

In conclusion a Ti indiffusion process in $LiNbO_3$ has been found which allows subsequent electric field poling with a dynamic similar to that achievable in as received material, without the need of any crystal surface grinding. Our experiments also show that if Ti indiffusion were to be performed at higher temperatures in the same atmosphere, or at the same temperature in a different atmosphere, subsequent poling would have a significantly different dynamic and could not be completed. These findings are in agreement with the formation of an insulating thin domain inverted layer during Ti indiffusion which prevents subsequent poling. The proposed technique can be crucial for high yield wafer processing of integrated electro-optic modulators and frequency converters.

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