

Electric-field poling of Ti indiffused z-cut LiNbO₃ wafers without surface grinding

D. Janner (1), F. Lucchi (2), P. Vergani (2), S. Giurgola (2), and V. Pruneri (1,3)

1) ICFO -Institut de Ciències Fotoniques, Mediterranean Technology Park. Av. del Canal Olímpic s/n 08860 Castelldefels, Barcelona, Spain. valerio.pruneri@icfo.es 2) Avanex Corporation Sede Secundaria, San Donato Milanese (MI), Italy 3) ICREA -Institutió Catalana de Recerca i Estudis Avançats, 08010, Barcelona, Spain

Abstract: *We have achieved domain inversion in Ti indiffused z-cut LiNbO₃ substrates through electric field poling without the need of any grinding process after Ti indiffusion. This allows a simplified, high yield and inexpensive production of domain engineered electro-optic and nonlinear optical devices, which is needed for an industrially viable processing over the full wafer scale.*

Introduction

In ferroelectric materials (e.g. LiNbO₃), the second-order nonlinear optical properties (including electro-optic and nonlinear optic properties) are intrinsically related to the crystal orientation (also called poling orientation or domain) [1]. In particular one can purchase LiNbO₃ substrates up to 5" in diameter which are already poled during or after the crystal growth. These wafers thus present a single domain structures, i.e. the same c- or z-axis orientation over the whole volume. It is also known that by applying high electric fields (>20kV/mm) through appropriate electrodes across single domain LiNbO₃ 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 not periodic fashion [7], to produce high frequency narrow band modulation and low driving voltage, respectively.

For both integrated nonlinear frequency conversion and electro-optic devices one needs also a suitable means to produce waveguides. The two most common techniques to produce waveguides in LiNbO₃ 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. The

worldwide market for integrated electro-optic modulators in LiNbO₃ is steadily increasing and >50000 units are produced each year. It has been recently shown that the performance of integrated electro-optic modulators in Ti indiffused z-cut LiNbO₃ can be significantly improved by making use of domain inversion. In particular, modulators with driving voltage well below 3 V [7,8] for inexpensive electro-absorption drivers as well as chirp free modulators [9] can be produced. It becomes crucial that Ti indiffusion is fully compatible with electric-field poling for an industrially viable production of wafers with a sufficiently large quantity of chips. In this paper we provide a receipt for Ti indiffusion which allows subsequent poling without the need of grinding one of the crystal surfaces after waveguide fabrication. In particular we show how the setting of Ti indiffusion temperature and atmosphere is critical to avoid the grinding process, which is unpractical for industrial wafer processing, time consuming and introduces high risks of breakage.

Experimental results

It is known that Ti indiffusion is accompanied by Li₂O outdiffusion from the crystal surface [10]. If ad-hoc measures are not taken to prevent Li₂O outdiffusion, domain inversion on the z+ crystal surface may occur: 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 [11]. On the contrary Li₂O outdiffusion does not cause any domain reversal in proximity of the z- surface and this is why 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 [12]. This is most likely related to the fact that the thin layer functions as an insulating layer for the poling. In

fact 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 is to be inverted. The presence of a thin layer which is already oriented as the applied field and at the same time insulating does not allow any current to reach the z+ surface of the initial crystal, which thus does not invert. An obvious 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 z-surface [5,12]. After removal of this thin layer the crystal can be poled. Beside being time consuming and expensive the grinding 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 and loss of all the chips.

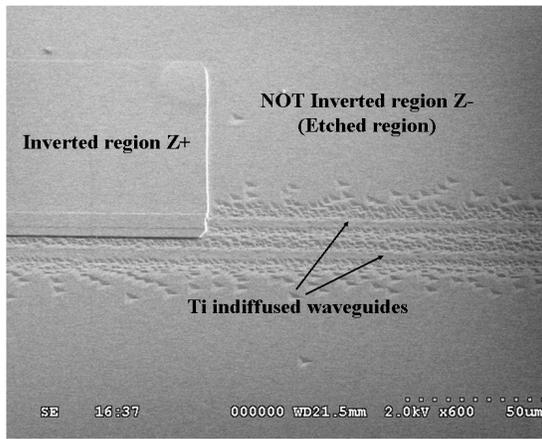


Fig. 1: Domain inversion in a Ti indiffused LiNbO₃ crystal revealed by etching: the domain inversion boundary is clearly in the middle of two optical waveguide Mach-Zehnder arms.

Contrary to what has been reported in the literature so far, our waveguide fabrication process does not prevent subsequent poling. After deposition of Ti metal strip on the z- face of a 0.5 mm 3" wafer, Ti indiffusion is performed at 1030°C for 9 hrs under an O₂ flow of 1.3 l/m. During the indiffusion dummy LiNbO₃ wafers are added to enrich the atmosphere with Li. Without any grinding process the wafer was 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 were domain inverted without any significant difference with respect to wafers as received (not indiffused

with Ti). Fig. 1 shows a typical result of a structure with Ti indiffused waveguides and domain inversion obtained by the above procedure.

Our belief is that the simultaneous use of low temperature (1030 °C) and O₂ atmosphere avoids the need of any grinding. The low temperature itself and O₂ enriched atmosphere prevent Li₂O out-diffusion. At the same time a lower temperature (i.e. a temperature further 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 conclusions are supported by poling measurements carried out on 4 different samples from the same wafer: (#1) crystal as received, no Ti indiffusion; (#2) crystal with Ti indiffusion at 1030°C, 9 hrs, 1.3 l/min O₂ flow and Li enriched atmosphere; (#3) crystal with Ti indiffusion at 1030°C, 9 hrs, 1.3 l/min N₂ flow and Li enriched atmosphere; (#4) crystal with Ti indiffusion at 1100°C, 9 hrs, 1.3 l/min O₂ flow and Li enriched atmosphere.

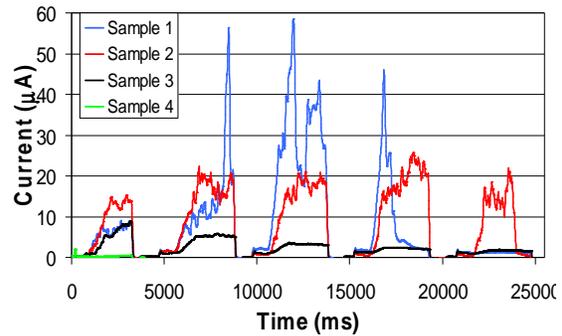


Fig. 2: Current during poling for samples: #1 (as received), #2 (1030°C, O₂ flow), #3 (1030°C, N₂ flow) and #4 (1100°C, O₂ flow).

The 4 samples have a quite different poling behavior (figs. 2-3). Fig. 2 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, O₂ flow) show a faster dynamic than #3 (1030°C, N₂ 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. 3 where it is evident that #1 completes poling (reaches 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 requested to invert the whole area (same as #1 and 2).

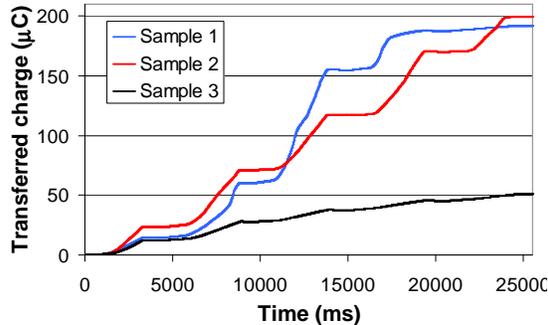


Fig. 3: Cumulative poling charge for #1, 2, 3.

Sample #4 (1100°C, O₂ flow) shows already a very low poling current (<0.5 μA) during first poling pulse at 10.5kV (fig. 2). To see whether poling efficiency could be increased at higher voltage pulses subsequent poling was performed at 10.7, 10.8, 10.9, until breakdown occurred at 11 kV. Even for these higher voltages the poling current was always limited to <3 μA , showing the impossibility to get significant poling in this sample (see fig. 4). Note that also, similarly to #4, also #2 above was subjected to higher voltage poling after the 5th pulse at 10.5kV without getting any significant increase in current (cumulative charge).

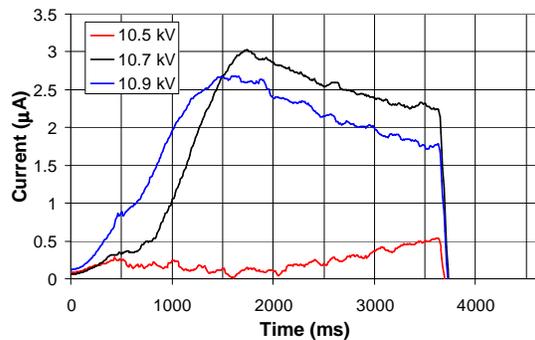


Fig. 4: Poling current of # 4 at increasing applied voltages where it is evident the negligible poling current even for higher voltages (before breakdown).

Conclusions

A Ti indiffusion in LiNbO₃ process 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 cannot 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 is crucial for high yield wafer processing of integrated electro-optic modulators and frequency converters.

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