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### Electric-field-induced periodic domain inversion in Nd<sup>3+</sup>-diffused LiNbO<sub>3</sub>

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*Indexing terms:* Lithium niobate, Electro-optical effects, Crystal waveguide lasers

The authors report the first demonstration of electric-field-induced periodic poling in neodymium-indiffused lithium niobate. The fidelity of patterning is as good as with undoped samples, and the poling field required is smaller. This novel combination of two techniques for manipulation of the optical properties of lithium niobate makes possible an extended range of active devices.

**Introduction:** Lithium niobate is an attractive material for optoelectronics applications, both in bulk and integrated devices. Its versatility has been enhanced recently by the demonstration of various techniques for crystal domain engineering, notably domain inversion induced by pulsed-*E*-field with patterned electrodes [1]. Such techniques may be used to create periodically poled crystals for a large range of functions, including quasi-phase-matched (QPM) structures for enhanced nonlinear optical and electro-optic interactions, and high-frequency modulation. In a parallel development, the diffusion of a variety of rare-earths into lithium niobate has been demonstrated, leading to high-quality active integrated optical devices including amplifiers and tunable lasers operating at room temperature, with output powers up to tens of milliwatts [2, 3]. It is a natural next step to combine the two techniques so as to make feasible such devices as efficient QPM intracavity-doubled lasers with output at visible wavelengths.

It is well known that the stoichiometry of lithium niobate can affect its domain structure [4] or the ease with which it can be poled. Moreover, the process of rare-earth indiffusion requires high-temperature treatment of lithium niobate over extended periods, which renders the final result very sensitive to details of the composition of the processing atmosphere. Thus there is no *a priori* assurance that rare-earth diffusion and *E*-field-induced domain inversion can be carried out on the same substrate. We report here the successful production of a periodically domain-inverted structure in Nd-diffused lithium niobate, proving for the first time the compatibility of these two processes.

### Experiment:

(i) *Nd diffusion:* We used 76mm-diameter 200µm-thick undoped Z-cut lithium niobate wafers provided by Mitsui Kinzoku (Japan). The wafers were diced with a diamond saw, into samples approximately 12mm × 12mm in size, and cleaned. 13±2 nm of Nd metal was deposited on the -Z face by vacuum evaporation from a tungsten boat. The Nd was then diffused into the lithium niobate by placing the samples in an alumina box in an atmosphere of dry oxygen for 240 h at a temperature of 1090°C. To reduce the out-diffusion of Li<sub>2</sub>O, a mixture of lithium carbonate (2.4 wt%) and lithium niobate powders was also placed in the box to raise the Li<sub>2</sub>O partial pressure. The Nd diffusion was thus carried out under standard conditions, appropriate for the fabrication of a waveguide laser, known to lead to diffusion depths of the order of 5µm and maximum concentrations of ~0.5 mol% [3].

(ii) *Removal of out-diffused layer:* Our initial attempts to pole Nd-diffused lithium niobate were unsuccessful. Polishing and room-temperature etching in 1:2 HF:HNO<sub>3</sub> of a Y-cut cross-section of the diffused samples revealed a shallow domain-inverted region on the undoped +Z face, presumably due to out-diffusion of Li<sub>2</sub>O [4]. This domain-inverted layer blocked the *E*-field poling. While we have succeeded in reducing the extent of this out-diffused layer by controlling the atmosphere during diffusion, as described above, we have not yet entirely eliminated it. An examination of a sample diffused under the conditions described above showed a residual domain-inverted layer about 7µm thick, pierced by numerous pinholes of the original polarity. For the experiment reported in this Letter, therefore, we had to eliminate the domain-inverted layer prior to poling. This was done by removing about 40µm of material from the +Z face, using a 3µm alumina/water lap followed by polishing with an aqueous suspension of silica. Note that the polishing removed material only from the undoped face of the sample.

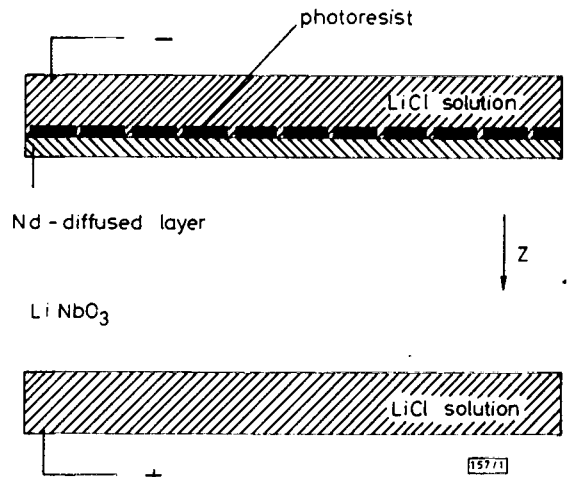


Fig. 1 Diagram of poling configuration

(iii) *Periodic poling:* The sample was poled as previously described [5] using filter paper soaked in aqueous lithium chloride solution as the electrodes, and 1.5µm of Shipley AR 1400-27 photoresist as an insulator to pattern the electrode on the doped, -Z surface. We used standard photolithographic techniques to open a comb-shaped window in the photoresist. The 'teeth' of the comb were 2mm long and 4µm wide, separated by strips of photoresist 16µm wide. The experimental configuration for poling is shown in Fig. 1. Poling of a neodymium-diffused area was achieved in a single 7.5ms pulse at 2.6kV. The time dependence of the poling current is shown in Fig. 2. It consists of an initial capacitive spike followed by an erratic current associated with the growth of inverted domains in the crystal.

(iv) *Sample characterisation:* To demonstrate the successful combination of the two techniques, we characterised the processed sample for neodymium doping and domain patterning. We confirmed the presence of neodymium by demonstrating unguided fluorescence in the 1100nm wavelength region from the Nd-doped area when it was illuminated with about 250mW of radiation at 809nm from a Ti:sapphire laser, using a setup similar to that described previously [6].

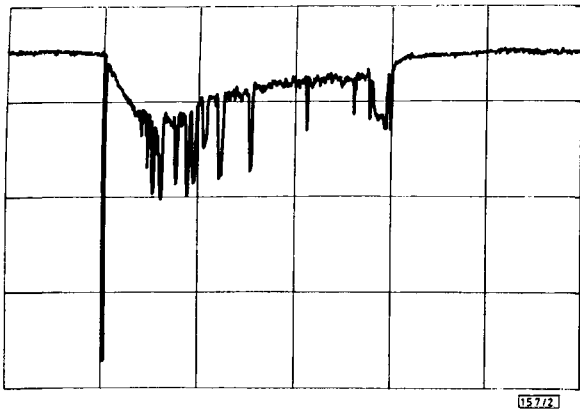


Fig. 2 Poling current measured during application

Vertical range : 8 mA; horizontal range : 15 ms.  
Initial spike is capacitive charging of substrate

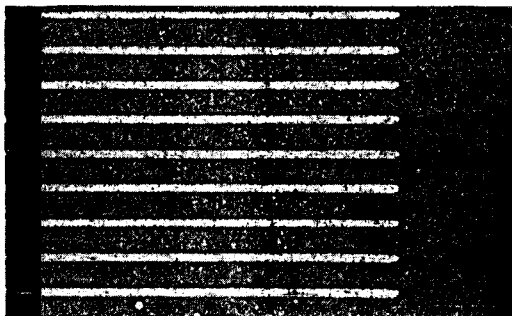


Fig. 3 Etched Nd-doped surface showing domain structure

Lighter regions are inverted. +Z domains

The domain-inverted regions were exposed by etching, and a typical example of the pattern on the Nd-doped surface is shown in Fig. 3. The inverted regions are the bright areas, as the +Z faces have not been roughened by etching. The inversion was almost complete and uniform over the entire area of the comb, with the exception of some small patches which we attribute to air bubbles in the electrode. The measured charge transfer of  $7\mu\text{C}$  is consistent with this degree of inversion. The mark/space ratio of the domain patterning shows a widening of the inverted regions by about  $1\mu\text{m}$  at each edge consistent with what is seen on undoped samples.

**Discussion:** We have demonstrated high-quality periodic domain-inversion in a neodymium-diffused sample of lithium niobate. In terms of the fidelity of pattern replication, there is no observable difference between doped and undoped samples. The field strength required for poling,  $17\text{kVmm}^{-1}$ , is substantially lower than the poling fields of about  $23\text{kVmm}^{-1}$  required for virgin samples of lithium niobate [5]. The reasons for this reduced poling field are currently being investigated.

Because the presence of the neodymium appears to have no deleterious effects on the poling process, we are confident that the period of the grating can be reduced to the  $6\mu\text{m}$  range required for first-order QPM for frequency doubling of light at  $1064\text{ nm}$  wavelength. We have already poled undoped samples with a  $3\mu\text{m}$  period. In a less demanding application, longer domain periods have the potential for suppression of photorefractive damage, as has been demonstrated in bulk  $\text{LiNbO}_3$  [5].

The samples we have used are rather thin, although not impractically so, and they have been thinned further by the need to polish the +Z surface. We note that samples up to  $0.5\text{ mm}$  thickness have been successfully poled [7], and we expect that further improvements in the conditions during the lengthy Nd indiffusion will obviate the necessity of polishing.

We are currently extending this process to the fabrication of a QPM self-frequency-doubled waveguide laser and to the demonstration of the suppression of photorefractive damage in waveguides.

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### High temperature CW operation of GaAs/AlGaAs high barrier gain offset VCSELs

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*Indexing terms:* Aluminium gallium arsenide, Gallium arsenide, Vertical cavity surface emitting lasers

Greater than  $150^\circ\text{C}$  CW lasing is achieved from an unbonded GaAs/AlGaAs VCSEL by employing high barrier confinement spacers and by blue shifting the optical gain. This is done while maintaining a variation in threshold current of only  $\pm 0.93\text{ mA}$  over a range greater than  $150^\circ\text{C}$ . These results are compared to a low barrier VCSEL of similar design.

Considerable effort has been devoted to the development of vertical cavity space emitting lasers (VCSELs). Many features including low threshold currents, high quality low diverging outputs as well as the ability to fabricate two dimensional arrays make VCSELs potentially attractive for many applications such as optical interconnection, communication and computing. Improvements in temperature performance are pursued to provide predictable operation in various thermodynamic environments.

Recently, methods for extending the high temperature operation of various VCSEL structures were examined [1-3]. It was shown that improvements in the temperature performance could be realised by: employing a high barrier confinement spacer to reduce the leakage currents at high temperatures; offsetting the gain peak with respect to the Fabry-Perot transmission peak at room temperature to provide more gain at higher temperatures; reducing the VCSELs self-heating by decreasing the series resistance of the semiconductor distributed Bragg reflector (DBR). Young *et al.* reported [1]  $145^\circ\text{C}$  CW operation of an unbonded VCSEL operating at  $\sim 997\text{ nm}$ . This bottom emitting structure contained a three InGaAs quantum well active region surrounded by a high barrier  $\text{Al}_0.5\text{Ga}_{0.5}\text{As}$  confinement spacer and GaAs/AlGaAs top and bottom DBRs where the interfaces were graded over  $18\text{ nm}$  to reduce the series resistance. The offset between the Fabry-Perot transmis-