REPORT DO JMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	Dec. 30, 1994	Final / May 15, 1994 - Nov. 14, 1994

4. TITLE AND SUBTITLE

Doubled Diode Laser

5. FUNDING NUMBERS

~~C: F49620-94C-0039~~

~~PR: 0840~~

65502F

3005/SS

6. AUTHOR(S)

Dr. David Deacon

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Deacon Research
2440 Embarcadero Way
Palo Alto, CA 94303

8. PERFORMING ORGANIZATION REPORT NUMBER

AFOSR-TR- 95 0122

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

USAF, AFMC
AFOSR/PKA
110 Duncan Ave. Ste. B115
Bolling AFB, DC 20332-0001

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

F49620-94-C-0039

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release;
Distribution Unlimited

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

In this Phase I program, we have demonstrated that electric field poling is an advantageous technique for manufacturing frequency doublers. We demonstrated bulk periodic poling with 15 micron period lithium niobate samples, and showed that we can overcome the problems with electrical breakdown. From our other testing results, we can predict that our periodic poled samples will perform near the theoretical optimum. While the poling process shows peculiarities such as a strong crystal axis dependence and a complicated voltage-time scaling, we conclude that the process is ready for development into a reliable manufacturing process.

14. SUBJECT TERMS			15. NUMBER OF PAGES
			16. PRICE CODE
17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT

FINAL REPORT

DOUBLED DIODE LASER

SBIR Phase I Contract F49620-94-C-0039

Period: May 15 1994 - November 14 1994

principal investigator:
David A. G. Deacon

Deacon Research
2440 Embarcadero Way
Palo Alto CA 94303
(415) 493-6100
FAX: (415) 493-6493

sponsor:
Air Force Office of Scientific Research
110 Duncan Ave. Suite B115
Bolling AFB DC 20332-0001

technical monitor:
Dr. Howard Schlossberg

AFBLUE1.WPD December 30, 1994

TABLE OF CONTENTS

Abstract

In this Phase I program, we have demonstrated that electric field poling is an advantageous technique for manufacturing frequency doublers. We demonstrated bulk periodic poling with 15 micron period lithium niobate samples, and showed that we can overcome the problems with electrical breakdown. From our other testing results, we can predict that our periodic poled samples will perform near the theoretical optimum. While the poling process shows peculiarities such as a strong crystal axis dependence and a complicated voltage-time scaling, we conclude that the process is ready for development into a reliable manufacturing process.

1. Background: Selection of Approach

There are a multitude of reasons for our selection of our approach, but the single most constraining criterion is cost. To commercialize lasers in volume, the cost must be reduced to the maximum extent possible. The more the cost is reduced, the larger becomes the range of applications. For example, to supply the need for blue lasers for data storage, a laser with cost below about $10 is required. It is our goal to meet this need.

Our conceptual system geometry is sketched in figure 1. This figure shows two chips with butt coupling as essentially the entire equipment needed to produce a laser. The first chip is a commercially available infrared GaAs-based diode laser, and the second chip is our quasi-phasematched (QPM) frequency doubler. In fact, we now prefer a planar flip-chip coupling geometry, but the exact geometry of the two chips is not the main point here. The main point is that once the QPM chip is available, we will have a revolution in laser applications due to the cost reduction implicit in a two-chip configuration. If both chips can be mass produced, a packaging technique can surely be worked out by which the two chips can be automatically assembled with proper alignment in high volume. The critical step is to demonstrate the QPM chip. This is our contribution.

Clearly, blue lasers must be mass produced in very large quantity (perhaps millions per month) to reach this cost. Our technology, by virtue of its photolithographic approach to manufacturing, is inherently capable of being scaled to this kind of volume. The material the laser is made from must be available in wafers and have low cost. Lithium niobate is our preferred candidate in both of these respects, and it has the highest nonlinear coefficient as well, so it is an obvious candidate to start with. KTP is far too expensive for this application, but $LiTaO_3$ is an interesting backup material if lithium niobate were to exhibit some unknown and insuperable problem.

Of course, not only blue lasers, but also green and ultraviolet lasers can be made with this technology. The blue and UV lasers will be used for high density optical hard drives, and the green and blue lasers in combination with existing red diodes will be used for RGB displays.

It is possible that a technology breakthrough in direct lasing blue diodes (e.g. ZnSe or GaN) may threaten our business strategy, but we also threaten the direct blue diodes. There are major materials science problems to be overcome by both the ZnSe technology (lifetime) and the GaN technology (threshold), and it is not predicted that they can approach our level of development for five years or more. By that time, we expect to be in full production. Since our projected manufacturing costs are comparable to projected direct diode manufacturing costs, there may be little reason at that time to tool up for manufacturing direct blue diodes.

Our technology also has important advantages over the various blue laser technologies. By frequency doubling the well-developed infrared-to-red range of GaAs based diode lasers, we can produce inexpensive laser light everywhere in the visible from the red into the ultraviolet. This wavelength range is much broader than that of any single blue laser technology. Our range also covers all the major market needs, from the red, green, and blue needed for displays, to the blue and ultraviolet needed for the evolution of high density optical disk drives over the next fifteen years. As a result of the flexible wavelength range of our doubled diode laser strategy, we can also be sure we have the capability of addressing other important applications even if the precise wavelengths are not known today.

Previous processing technology of lithium niobate (e.g. by Ti indiffusion) is not capable of efficiently generating the required blue power due to low efficiency and photorefractive damage problems [1]. The low efficiency is due to intrinsic problems with angled domains which are solved by electric field poling. It has also been shown

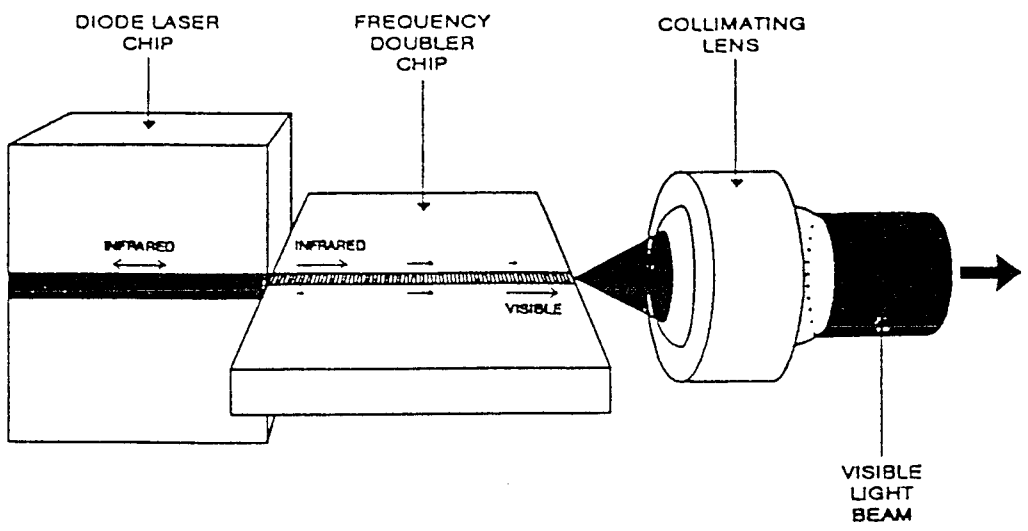


Figure 1

Conceptual diagram of QPM waveguide doubled diode laser.

that the high photorefractive sensitivity of these devices probably limits the output power to not much more than a milliwatt or so [1]. Electric field poling is also known to relieve the photorefractive problem: 20 mW has been demonstrated by Sony with no degradation. With E-field poling, $LiNbO_3$ has shown the highest conversion efficiency (600%/W-cm^2) of any waveguide doubler material, and it has produced the highest blue output power (20 mW) without laser damage [2]. The results of this Phase I project combined with our internal work at Deacon Research (see the Related Work section below) have shown similar results and we have extended their results to bulk poling.

2. Phase I Results

2.1 Demonstration of 15 μm Bulk Periodic Poling

We have demonstrated electric field induced domain inversion of z-cut $LiNbO_3$ with a period of 15μm. (The term domain inversion refers to the reversal of the direction of the crystalline z-axis in desired areas of a crystal.) The inverted domains pass through the entire thickness of the 500μm crystal wafer creating a substrate usable for both waveguide and bulk optical interactions. Much of this work uses proprietary processing technology which we have developed through our own internal research programs. In what follows, this proprietary technology is not described, but we provide form fit and function data instead. For another project [3], we also had the opportunity to produce samples at the longer period of 29.3μm. We were able to delay the technical start of this program so as to benefit from both our internal work and the results of the prior project. As a result we have made extremely rapid progress here.

The transition to a 15μm period required the determination of a new set of poling conditions. The problem which must be solved is to fill out the required domain pattern across the desired grating without allowing the domain grating lines to merge together. This requires a trade-off to be reached between the poling parameters of pulse voltage, length and poling current as outlined in Table I. Patterned domain inversion in z-cut $LiNbO_3$ is achieved by applying a spatially varying electric field greater than the crystalline coercive field. The electric field must oppose the spontaneous polarization of the crystal sample, i.e. a negative high voltage pulse must be applied to the -z face of the crystal. The experiments reported here used a liquid conductor to achieve electrical contact with the +z and -z surfaces of the crystal. The conductor of choice for our experiments is an aqueous solution of lithium chloride. The concentration of the

pulse parameter	too high	too low
pulse voltage	increased chance of destructive sample breakdown	patchy domain seeding, grating incompletely formed
pulse length	domains merge together to destroy grating pattern	domain grating incompletely formed, poor uniformity
poling current	increased chance of sample breakdown	domain grating incompletely formed, poor uniformity

Table I

Effects of Poling Pulse Parameters

solution does not seem to have any significant effect on the poling which can be achieved, and both saturated and very weak solutions have been used.

The experimental arrangement is shown in figure 2. A custom high voltage pulser owned by Deacon, was connected to a lithium niobate wafer through a current limiting resistor using liquid electrodes. The applied voltage and current are monitored and digitized on each pulse.

The spatial modulation of the electric field was created photolithographically using a mask to define an electrode pattern using a mask deposited on the + or - z

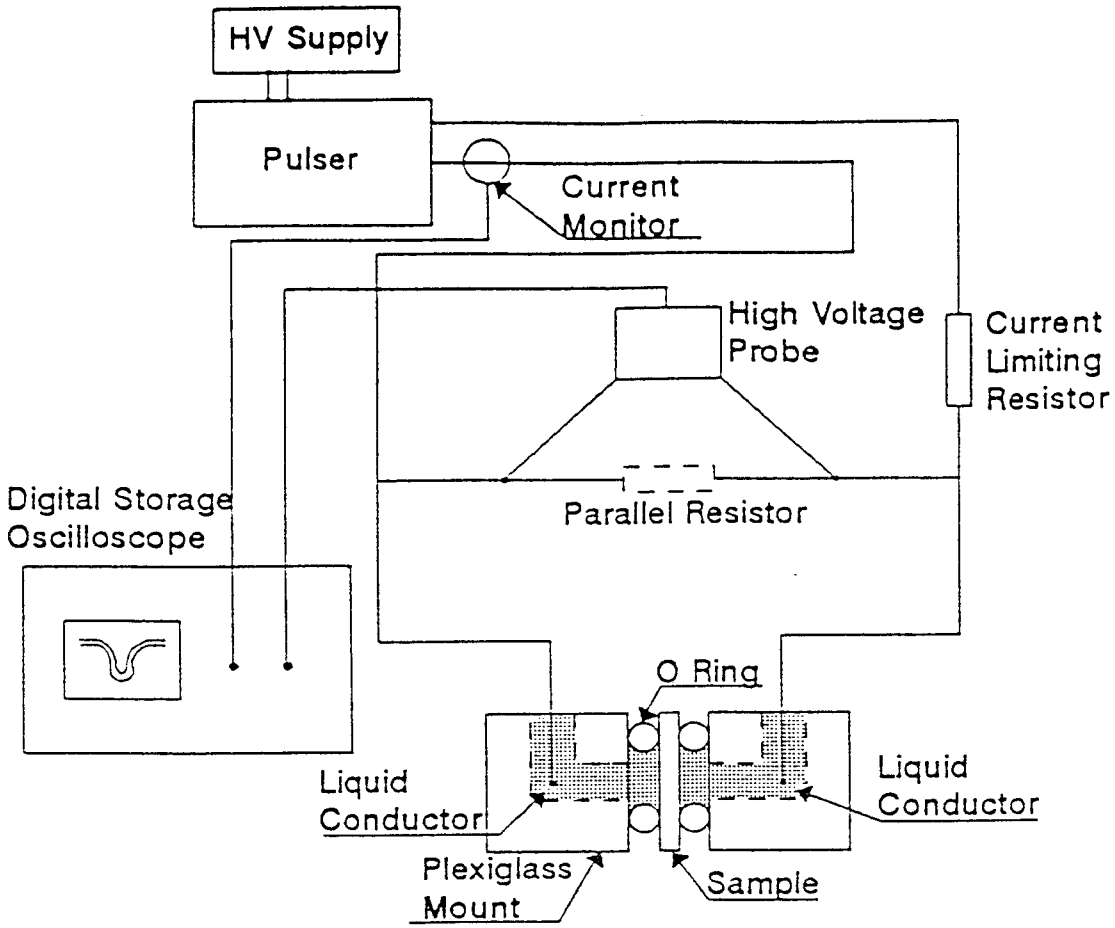


Figure 2

Schematic diagram of circuit used for E-field poling.

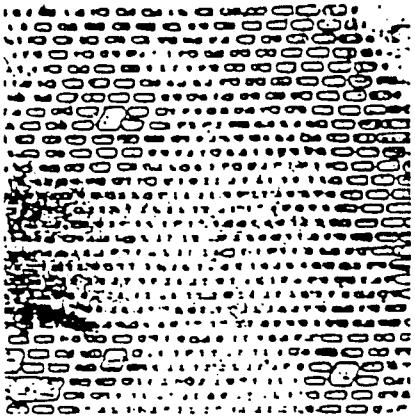
face. Reproduction of the mask pattern into the poled pattern was found to be most accurate when the insulator was applied to the +z face, although large features could be reproduced from a mask patterned on -z. The resist layer was patterned photolithographically using a chrome on glass photomask, and was developed and hard-baked. Lithography was performed on quartered 3" diameter wafer sections, and the individual samples diced after completion of the mask fabrication steps.

Initial experiments created only isolated patches of domain features which accurately reproduced the original mask dimensions. Other areas showed either under- or over-poling (merging of adjacent domains). This was ascribed to the seeding

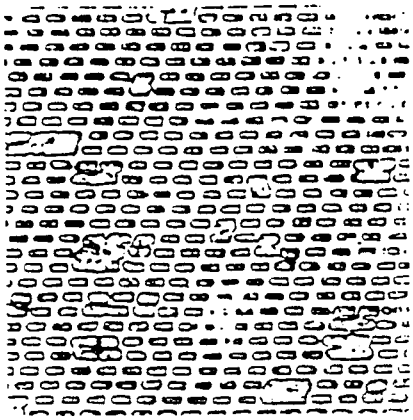
of domain inversion commencing at different times in different areas of the sample. Those areas which seeded early in the pulse would show complete patterned features before some areas started to pole at all. Such variation in seeding time could be the result of material inhomogeneities which raise or depress the localized poling threshold, or it could result from inconsistencies in the electrical contact to the sample via the liquid conductor. Whatever the cause, extending the pulse length sufficiently to achieve complete poling in the desired patterned areas leads to significant overpoling and merging of those areas which are seeded first.

The desired domain grating bars were formed from shorter segments arranged in rows as indicated in figure 3. The stagger of segments in adjacent grating lines was designed to allow characterization of the samples using a bulk frequency conversion experiment. Our initial poling results are illustrated in figure 3a, which is a region of the domain pattern on the +z face, created using a 40 μs pulse of 13.4kV amplitude, revealed by etching the crystal in hydrofluoric acid for ~10 minutes (+ and -z directions etch at different rates yielding a surface relief visible under the microscope). Domain inversion seeding is clearly visible, although the pulse length is clearly too short to complete the poling of all the features at this voltage level. Increasing the voltage at this pulse length to complete the patterned domain reversal is not desirable as destructive breakdown events increase in likelihood with higher voltage. Also, increasing the voltage too far will drive the domains laterally under the photoresist mask, merging those areas which are first to nucleate.

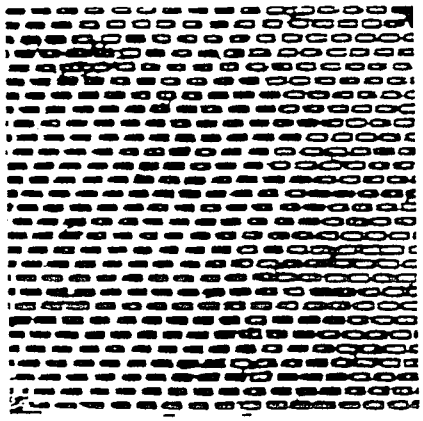
A more complete grating over a 5mm diameter sample was fabricated by applying two 40μs pulses of differing amplitudes. The first, 13.2kV pulse was designed to yield seeding similar to that shown in figure 3a. A second, 12.5kV pulse was applied to attempt to fill in the desired domain pattern without exceeding the threshold for poling under the mask and expanding the domain features past the desired borders. The domain pattern of this sample is shown in 3b and is significantly better filled than



3a. Photomicrograph of domain pattern on +z face showing seeding of segmented domains after a single 40 μs, 13.4 kV pulse.



3b. Domain pattern showing better filling of desired features after two 40 μs pulses: one at 13.2 kV and one at 12.5 kV.



3c. Domain pattern showing good uniformity after two 11.3 kV 125 μs pulses.

Figure 3
$LiNbO_3$ samples poled with15.8 μm period, etched to reveal domain reversal regions.

shown in 3a. There are still some areas which show only seeding, indicating that the applied pulses are still short of optimum, and there are also a few areas where the insulating mask failed to prevent lateral domain motion, and merging has occurred.

Decreasing the applied voltage and increasing the pulse length led to further improvements in uniformity as illustrated in figure 3c. Two pulses of 125μs duration and 11.3kV amplitude were applied, separated by about 30 seconds. A few domains have merged together in isolated areas of the sample possibly as a result of defects in the insulating layer composition or crystal inhomogeneities.

The three dimensional nature of the domain inversion pattern revealed by etching the +z surface can be assessed by cutting the crystal perpendicular to the y-axis followed by polishing and etching. During domain inversion the directions of both the z- and y-axes are reversed. The differential etching rates between the +y and -y axes is used to reveal the domain pattern in the cross section (the x axis is invariant under domain inversion, and does not exhibit differential etching between +x and -x). A photomicrograph of a cross section through a 15.8μm periodically poled crystal is shown in figure 4. The inverted domains stemming from a patterned mask on the +z face clearly propagate through the crystal parallel to the z-axis. In fact the domains pass completely through the crystal revealing a very similar pattern on the -z face to that of the mask on +z.

In very short order, we have been successful in producing periodic poled samples with reasonable uniformity at a 15 micron period. Although the duty cycle is not yet perfectly under control, we can say from our experience characterizing our 29 micron period samples that the sample shown in figure 4 can be used effectively for third order frequency doubling and should yield better than about 50% of the theoretical conversion efficiency. In the Option to the Phase I program, we will measure samples such as this one to determine the third order conversion efficiency.

2.2 Control of Electrical Breakdown

Electrical breakdown during poling can take either of two forms: the electrodes may discharge along a path around the outer surface of the sample, or along a path through the sample itself.

The most significant form of breakdown is the creation of a short circuit path through the crystal. In general this leads to shattering of the crystal substrate. The precise cause of these short circuit paths has yet to be identified, but it is likely that they are related to defects in the crystalline lattice. Crystal wafers which have been repeatedly processed through temperature cycles and the application of metallic surface coatings appear to be somewhat more susceptible than pristine crystals, perhaps due to the creation of stress-induced microcracks. Poling of crystals at high voltages and currents also typically leads to more breakdown events than poling at lower voltages and low currents.

Elimination of this destructive electrical breakdown can be achieved with the appropriate choice of poling parameters. For instance in a run of 65 samples investigating domain shape uniformity and the effects of small variations in pulse conditions, we observed only 2 destructive breakdown occurrences for a >95% yield. However, the yield depends strongly on the pulse parameters and we cannot yet predict this dependence. In another experiment with ≈12mm diameter samples, we observed ≥75% yield. The required set of pulse parameters for minimal breakdown will typically be different for each class of sample (e.g. sample size, surface treatment). Finding the correct parameter set requires minimizing the applied voltage and current while keeping the pulse length short enough so that lateral motion of the domain wall boundaries does not lead to merging of adjacent domains.

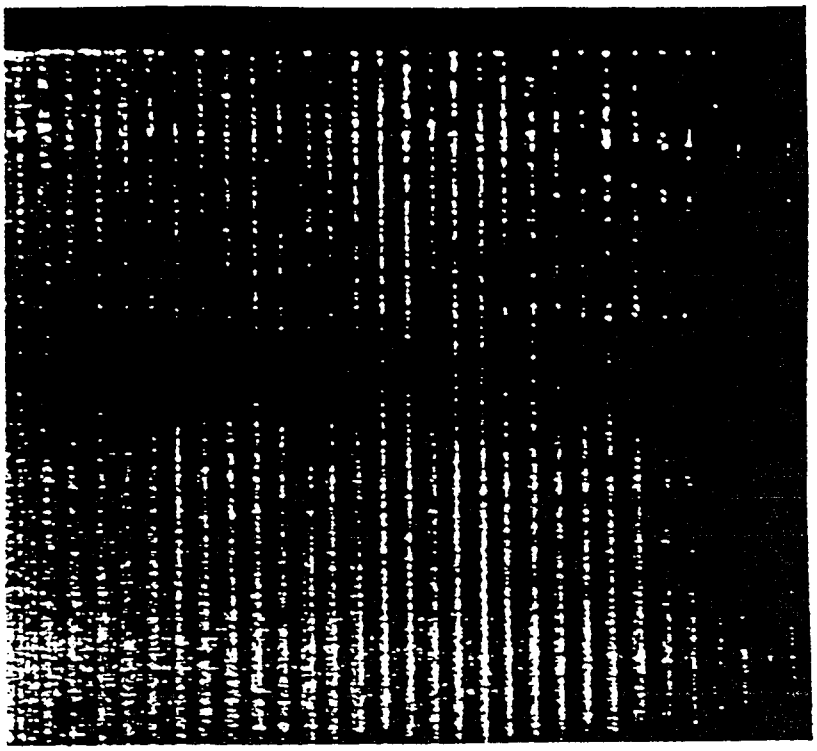


Figure 4
Cross section through a 15 μm periodically poled sample.

Figure 5a. Photomicrograph of 29.3 μm domain pattern on the -z face. Grating bars parallel to the y axis.

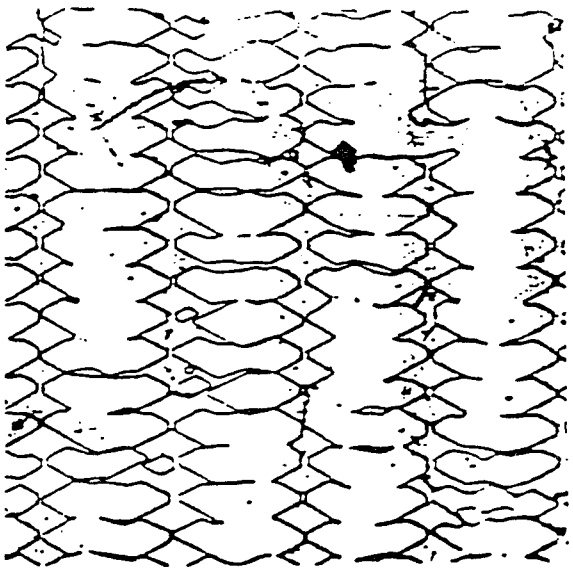


Figure 5b. Photomicrograph of 29.3 μm periodic domain pattern on the - z face. Grating bars parallel to the x axis.

Electrical breakdown along a surface layer of the sample, or between deposited layers can occur if the layer in question extends under the clamping o-ring to the edge of the crystal. Such surface breakdown burns the layer off the surface, destroying the usefulness of the sample. Immersing the sample in oil during poling is an expedient for eliminating surface breakdown, and we may yet use it in experiments at high voltage, but so far our work has not been particularly hampered by surface breakdown.

Breakdown through or around the o-rings clamping the sample typically does not physically affect the sample, which after cleaning can be reused and successfully poled. Such breakdowns are generally the result of either poor cleaning of the sample or o-rings before poling, or seepage of the electrolyte liquid under the o-rings during sample set-up. To provide maximum resistance to seepage soft silicone o-rings were chosen, maximizing the contact patch between crystal and o-ring, whilst keeping the clamping force low (applying too much clamping force can severely stress the crystal and lead to greatly increased chance of destructive breakdown). For large sample sizes (~12mm diameter), square section o-rings were found to perform significantly more consistently than standard round o-rings.

In order to eliminate breakdown completely as a factor in process yield we will need to more fully understand its causes. It would be useful to investigate the voltage dependance of breakdown frequency, as well as its dependance on surface layers and treatments. The role of material defects might also be analyzed to help predict the behavior of the breakdown probability with sample size.

2.3 Crystal Axis Dependence of Poling

When a high voltage poling pulse is applied to the surface of an unmasked $LiNbO_3$ crystal at room temperature, the resulting repoled domains are hexagonal in shape. The domain boundaries follow the crystal planes, with straight boundaries parallel to the crystal y-axes and its cousins inclined at 120°.

When a patterned insulating mask is deposited on the +z face of the crystal to provide patterned poling, the degree of replication of the mask pattern is dependent on the orientation of the mask with respect to the crystal. This is shown in the photomicrographs of figure 5 which reveal the domain patterns on the -z face after electric field poling of two samples using the same pulse parameters.

In figure 5a the domain grating bars are oriented parallel to the y-axis of the crystal, in 5b they are parallel to the x-axis. The shape of the inverted domains is clearly dependent on their orientation, with those parallel to the y-axis producing a relatively well defined grating, and those parallel to x- showing significant lateral wall motion and merging of adjacent domains. The lateral spreading of the x-oriented domains can be ascribed to the 'desire' of the inverted domain to have a boundary at 30° to the x-axis, as observed in bulk unpatterned poling, rather than parallel to it.

In these early days of our understanding of the poling process we can fabricate useful domain grating structures by ensuring that the long dimension of the grating bars is oriented along the y axis. As our understanding of the domain seeding process improves we expect to become able to choose suitable masking materials and pulse conditions to control the spread of the domains perpendicular to the x-axis so that arbitrarily oriented and shaped domains can be poled.

2.4 Voltage-Time Scaling of Poling

The threshold voltage for E-field induced poling has been found to be a function of the applied pulse length. When poling identically prepared and masked samples the threshold voltage increases as the pulse length is shortened. For a 125μs long pulse the poling threshold was 12.0 kV measured across the liquid contacts. With the pulse length expanded to 1ms the threshold for poling decreased to ≤11.2kV. This implies that there is some kind of a 'voltage-time product' which must be exceeded for poling to occur.

This result is supported by the experimental observation that for a fixed pulse voltage (close to threshold for the 1ms pulse) and identically patterned samples, a greater area of domain inversion is produced by a single 1ms pulse than by 10 consecutive 100μs pulses (applied at approximately 10 second intervals). Each of the shorter pulses is farther below the 'voltage-time product' threshold than the single longer pulse, so much less poling is observed.

Work is needed to investigate the trade-off between changing the pulse length and the voltage. Increasing the pulse length will increase the total lateral motion of the domain walls if the speed is constant, but decreasing the pulse voltage will decrease the lateral domain wall velocity. We will need to determine experimentally which way to play this tradeoff. We will of course have to watch out for potential increases in breakdown probability due to increased poling voltages associated with shorter pulses.

3. References

1. S.J. Field, M.M. Fejer, severe photorefractive damage effects appear at the 0.5 mW level of green generation, above which the normalized conversion efficiency drops rapidly; private communication, Stanford University, Palo Alto CA, 1993.
2. M. Yamada, N. Nada, M.O Saitoh, K. Watanabe, "First order quasi phase matched $LiNbO_3$ waveguide periodically poled by applying an external field for efficient blue second harmonic generation", Appl. Phys. Lett. 62 435-436 (1993)
3. Contract B235362 with Lawrence Livermore National Laboratory, Dr. Al Ramponi, contract monitor.