Appl. Phys. B 72, 301–306 (2001) / Digital Object Identifier (DOI) 10.1007/s003400100493

Applied Physics B
Lasers
and Optics

Periodic poling of magnesium-oxide-doped stoichiometric lithium niobate grown by the top-seeded solution method

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Received: 28 June 2000/Revised version: 12 September 2000/Published online: 7 February 2001 – © Springer-Verlag 2001

Abstract. Magnesium-oxide-doped stoichiometric lithium niobate has been produced using the technique of top-seeded solution growth from a lithium-rich melt. Optical tests, performed with a combination of argon-ion laser lines, have confirmed a previously published result (at 532 nm) that this material has superior resistance to photorefractive damage. This material has been shown, for the first time, to be amenable to periodic poling. Optical parametric oscillator tests have shown that this material maintains the advantages of periodically poled, congruent, un-doped lithium niobate while showing no evidence of photorefractive damage under typical operating conditions. Operating wavelengths as a function of quasi-phase-matching period and temperature have been measured for the optical parametric oscillator, providing useful new information about refractive-index dispersion in this material. This work establishes periodically poled, magnesium-oxide-doped stoichiometric lithium niobate as a viable material for nonlinear optics.

PACS: 42.70.Mp; 42.65.Yj; 77.84.Dy

Periodically poled lithium niobate (PPLN) has been developed into one of the most versatile nonlinear optical materials over the past 10 years. Electric-field poling has been the method of choice for engineering the material, as first demonstrated by Sony and perfected by several other groups [1–3]. Although PPLN (for the remainder of this paper, when we use the acronym 'PPLN', we assume that the lithium niobate has the congruent composition, with no magnesium oxide doping) has proven very useful in the frequency conversion of diode lasers and diode-pumped solid-state lasers because of its large nonlinear coefficient, its performance at high average powers can be limited because of two damage mechanisms, the photorefractive effect and green-induced infrared absorption (GRIIRA) [4, 5]. Proven methods for mitigating these effects create serious practical problems for PPLN-based devices because of the need to heat the PPLN crystal. Moreover, the ability of PPLN crystals subject to these damage mechanisms to achieve lifetimes comparable to those of the lasers which pump them has not yet been proven. In order for periodically poled nonlinear optical materials to gain wider acceptance, they must be produced in a form which is free of these damage mechanisms even when operated at room temperature. In this paper, such a material is demonstrated: periodically poled, magnesium-oxide-doped stoichiometric lithium niobate (PP-MgO:SLN). This material shows better resistance to photorefractive damage than the established material, PPLN, while retaining its advantages. In separate work, MgO:SLN has been shown to have greatly reduced GRIIRA in comparison with congruent lithium niobate (CLN) [5, 6], suggesting that the periodically poled version of this material will have superior resistance to damage caused by GRIIRA.

PPLN is produced from wafers of CLN. Because the congruent composition is lithium-poor (with a Li/Nb ratio of 48.6%/51.4%), the base material contains a large number of intrinsic defects. These intrinsic defect sites, and the impurities which occupy some of them, are considered to be major contributors to the photorefractive effect and green-nduced infrared absorption in the material [4]. In addition, the intrinsic defects also produce a high internal field which increases the size of the external field needed to produce domain inversion [7]. In principle, if the material were grown with perfect stoichiometry and purity, it might be expected that there would be no photorefraction or GRIIRA. A closely related material, lithium tantalate, has been shown to be free of photorefraction and GRIIRA when grown with nearly perfect stoichiometry [5, 8]. By contrast, lithium niobate grown with a nearly stoichiometric composition has proven to be more susceptible to photorefractive damage than congruent lithium niobate [9]. However, the damage in stoichiometric lithium niobate (SLN) can be eliminated by the addition of a small amount of MgO dopant to the melt [10], requiring a significantly smaller concentration (~ 1 mole %) than the amount needed to accomplish the same result in congruent material (5%) [11]. Because high levels of MgO doping typically lead to difficulties in the growth of high-quality crystals, MgO-doped SLN has been identified as a promising alter-

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native to CLN and MgO:CLN for nonlinear optical interactions [10]. The fact that MgO:SLN tends to be single domain as grown, and has a shorter absorption edge than CLN, adds to its appeal. Considerable effort has been dedicated to the development of a new crystal-growing technique, the double-crucible Czochralski method, which is capable of producing MgO:SLN in large volumes [9, 12].

Electric-field-periodic-poling of MgO:CLN has been reported to be significantly more difficult than poling of undoped CLN, raising concerns about the deleterious effects of magnesium doping on the ability to control domain merging at MgO concentrations high enough to eliminate photorefractivity [13, 14]. Although periodic poling of MgO:CLN has been carried out using the corona-discharge method, the area over which high-quality periodic poling could be achieved ($\sim 100\,\text{mm}^2$) was significantly smaller than the wafer-scale areas patterned using electric-field poling on CLN [15]. In this paper the production of PP-MgO:SLN is demonstrated for the first time using electric-field poling.

The remainder of this paper is organized as follows. First, production of MgO:SLN crystals using top-seeded solution growth is described. Next, results of optical damage testing are given. The results of experiments on patterning of this material using electric-field poling are followed by details of the performance of PP-MgO:SLN inside the cavity of an optical parametric oscillator (OPO), compared with that of a control sample of PPLN. A final section summarizes and concludes the paper.

1 Material growth

Several nearly stoichiometric un-doped and Mg-doped $LiNbO_3$ crystals were grown at Deltronic Crystal Industries using the top-seeded solution method and a melt with a lithium-rich composition (58% Li_2O, $[Li]/[Nb] = 1.38$). Transparent crack- and inclusion-free single crystals were produced with a slow pull rate of 0.3 mm/hr in the z-direction. An automatic diameter control (ADC) system was not employed since the growth process was slow, 0.2–6.0 g/hr. The distribution coefficient of Mg in the SLN crystals was found to be 0.9. The proportion of the melt solidified was limited to $0.1-0.2$ to ensure that the melt composition would not reach the eutectic composition (otherwise, the uniformity of the $[Li]/[Nb]$ ratio in the solid would have been compromised). The concentration of Mg in the solid was believed to be higher by a factor of 1.02 at the bottom of the boule than it was at the top of the boule because of the changing melt composition, assuming a constant distribution coefficient. Crystals weighing up to 1000 g were grown from 5000 g of melt. The novel double-crucible Czochralski (DCCZ) method with automatic powder-supply system [9] should be applied in practical production to grow homogeneous crystals with a high solidification ratio. Concentrations of Li, Nb and Mg in the grown crystals were analyzed using inductively coupled plasma atomic emission spectrometry (ICP-AES) [16]. It was found that the un-doped crystals grown from a lithium-rich melt exhibited a slightly larger value of the $[Li]/[Nb]$ ratio (0.993) than the 1 mole % Mg-doped crystals (0.986). The Curie temperatures of the grown crystals were measured using differential thermal analysis (DTA), and were found to be ∼ 1200 °C for the un-doped crystals and ∼ 1205 °C for the Mg-doped crystals. A picture of one of the MgO:SLN crystals is shown in Fig. 1.

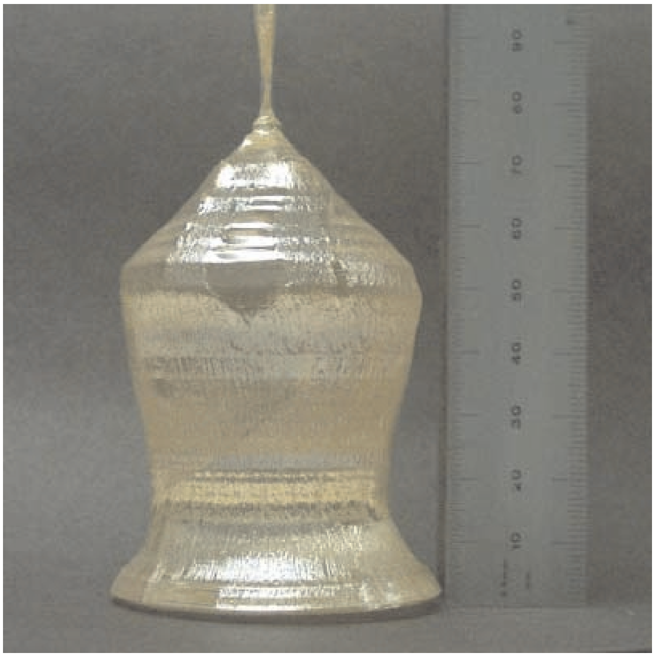

Fig. 1. Photograph of an as-grown boule of Mg-doped nearly stoichiometric $LiNbO_3$

2 Optical damage testing

Simple comparative experiments to measure resistance to photorefractive damage in single-domain material of different compositions were carried out at Gemfire Corporation. Radiation from a continuous-wave argon-ion laser operating in the all-lines configuration (Coherent Innova 90) was focused into samples with optically polished end faces. In the all-lines configuration, laser power appears at a variety of wavelengths, with most of the power at 514 nm and 488 nm. No filter was used. The spot sizes at the beam waist were approximately 20 μm (vertical) and 17 μm (horizontal). Testing was performed at room temperature with no external heating or cooling of the crystal. The output beam mode shape was monitored using a Spiricon beam analyzer. The output power of the laser was set to a few μW and slowly increased. At low power levels, the output beam mode shape was the same as the input mode shape as there was little or no photorefractive effect. As the power increased, the photorefractive effect distorted the beam shape, causing it to fan out in the z-direction. The power threshold for beam fanning was used as a comparative measure of the photorefractive response of that material. In some cases, no damage was observed at the highest power levels which could be produced with the argon-ion laser. The results are summarized in Table 1.

Table 1 shows that the threshold for photorefractive damage in MgO:SLN is at least three orders of magnitude larger at a mixture of argon-ion laser wavelengths than the corresponding threshold for un-doped CLN. The table also shows that MgO:SLN is superior to MgO:CLN under these test conditions, because the latter material began to show damage at the highest power level used in this test. These results can

Table 1. Observed thresholds for photorefractive beam distortion in a variety of materials from different suppliers

Material [supplier]	Power for fanning (mW)	Intensity for fanning (kW/cm^2)
CLN [Crystal Technology]	2	0.37
5% MgO:CLN [Yamaju]	1860[a]	350
SLN [Deltronic]	10	1.9
1% MgO:SLN [Deltronic]	> 1860[b]	> 350
CLT[c] [Yamaju]	15	2.8

[a] The observed beam distortion was the formation of a donut-shaped mode.

[b] No beam distortion was observed at the highest power used in the test.

[c] Congruent lithium tantalate.

be compared qualitatively with those reported by Furukawa and co-workers, who used a similar technique (visual observation of beam fanning) but a different laser wavelength (532 nm) [10]. The orders-of-magnitude superior damage resistance of MgO:SLN relative to SLN and CLN, shown in Table 1, is consistent with the earlier work. The slightly higher damage resistance of SLN relative to CLN, shown in Table 1, is not consistent with the earlier work. Further study of damage threshold as a function of laser wavelength is needed to resolve this discrepancy.

3 Periodic poling

Periodic poling of SLN and MgO:SLN was performed at Gemfire Corporation using a modified version of Gemfire's previously established process for patterning CLN [2].

Poling experiments were performed on 0.5-mm-thick, 50-mm-diameter wafers of SLN and 1% MgO:SLN cut from the boules grown at Deltronic Crystal Industries. The coercive fields of the materials were first determined using the technique of applying a slow ramping voltage to an unpatterned sample at room temperature, with electrical contact to the bare crystal $\pm z$-surface achieved using LiCl:H_2O liquid-electrolyte contacts. The coercive field was determined from the voltage at which a measurable current flow was observed in the poling circuit, scaled to the thickness of the wafer. The coercive field for un-doped SLN was determined to be 6.6 kV/mm, in reasonable agreement with a previously published measurement [7]. For MgO:SLN, a value of ~ 3.5 kV/mm was determined. This is dramatically lower than the coercive field for CLN, which was measured to be 21 kV/mm using the same technique. From the observed current flow and the measured poled area, the charge transfer required to achieve domain reversal was determined to be very similar to the value for CLN, $\sim 1.56\ \mu C/mm^2$.

To prepare a sample of MgO:SLN for periodic poling, a grating pattern was photolithographically defined on the $+z$-face of a wafer using photoresist. The pattern contained the following quasi-phase-matching (QPM) periods: 28.2, 28.5, 28.7, 28.9, 29.1, 29.3, 29.5, 29.7, 29.9, 30.2, 30.4, 30.6, 30.8 and 31.0 μm. The pattern was oriented with the grating lines parallel to the y-axis [2]. Care was taken to minimize thermal shocking of the MgO:SLN during processing, to minimize the introduction of defects that can otherwise distort the poled pattern [2]. A metal coating (NiCr/Au) was deposited on top of the patterned photoresist, and electrically contacted to a metal electrode using filter paper soaked with a saturated aqueous solution of LiCl. The $-z$-face of the wafer (which was not metallized) was similarly contacted. The metal electrodes, in turn, were connected to a computer-controlled high-voltage power supply. The applied voltage was monitored by a high-voltage probe (Tektronix P6195 A) and displayed on a digital oscilloscope (LeCroy 9314). Current flow in the circuit was monitored from a series-resistor chain and sampled by an analog–digital converter, whose output was transferred to the computer for control of the pulse length.

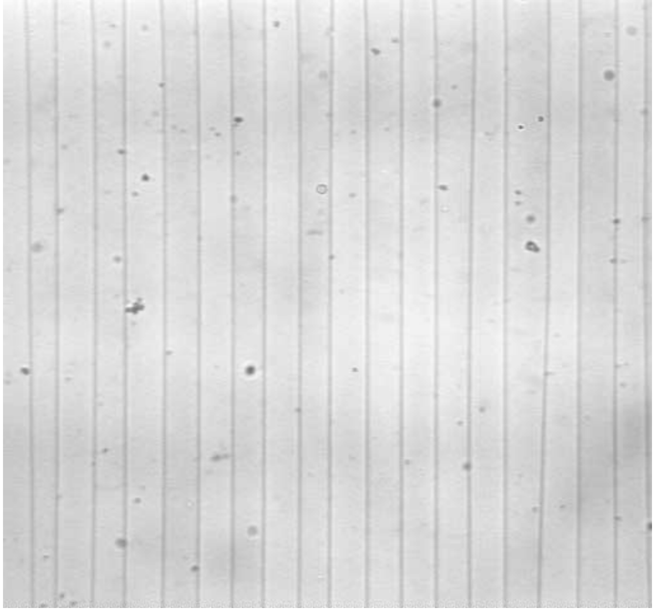

Fig. 2. Photograph of a patterned structure on MgO:SLN, with a QPM period of 29.3 μm. This view shows the $-z$-surface which had the uniform electrode

A single high-voltage pulse of ~ 2 kV was applied, and automatically shut off under computer control when the appropriate amount of charge had been transferred to the crystal, after about 100 ms. After the wafer was poled, the domain quality was characterized by etching in HF for about 5 min. Differential etching in the $\pm z$-directions revealed the QPM grating, as shown in Fig. 2. A 5-mm-wide sample with a length of 2 mm (having good poling quality over $\sim 90\%$ of its surface) was cut from the wafer and polished for optical testing.

4 OPO testing

The PP-MgO:SLN sample was tested in an OPO cavity at Lightwave Electronics. For comparison, a control sample of PPLN (prepared at Lightwave Electronics), of the same length, with a similar range of QPM periods, and containing the same anti-reflection coating, was tested in the same OPO cavity.

The OPO was pumped by a Q-switched neodymium yttrium–aluminum-garnet (Nd:YAG) laser at 1064 nm with pulse length of 25 ns at 10 kHz, as shown in Fig. 3. For the OPO resonator, a two-mirror linear cavity was used with the cavity mode waist centered in the nonlinear crystal. The

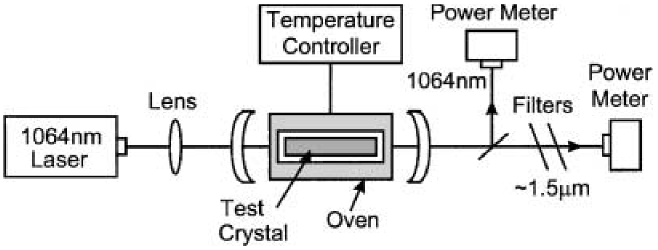

Fig. 3. Schematic diagram of the optical parametric oscillator used for sample characterization. The OPO was pumped by an acousto-optically Q-switched Nd:YAG laser at 1064 nm, while the crystals tested were PP-MgO:SLN and PPLN

input mirror was highly reflecting at the signal wavelength (1440–1640 nm), while the output coupler was 90%–94% reflecting over the same range. Two sets of experiments with different focusing conditions were carried out. In the first set of OPO experiments, the pump laser ($M^2 = 1.05$) was focused to a spot size of 53 µm in the center of the crystal. In the second set of OPO experiments, the pump laser spot size was 42 µm. Lifetime testing (operation over many hours at a constant pumping level) was not carried out in either case.

The first set of OPO experiments was carried out on a PP-MgO:SLN crystal without any anti-reflection coating. Average pump powers as high as 400 mW (several times the observed OPO oscillation threshold) were used. The high gain of PP-MgO:SLN and the high peak power of the Q-switched pump laser made oscillation possible in spite of the 14% reflection loss on each surface of the crystal. (Similar PPLN OPOs using un-coated crystals have been operated with great success [17].) At 150 °C, operation at signal wavelengths between 1440 nm and 1600 nm was obtained by varying the grating period, which was reasonably close to the operating wavelengths for a PPLN OPO with a similar set of periods [17]. The crystal experienced surface damage before quantitative data could be obtained. The maximum surface fluence of 0.45 J/cm^2 in a 25-ns pulse was below the commonly accepted damage threshold of 3 J/cm^2 in a 10-ns pulse for lithium niobate [18], but that threshold depends on the actual surface finish and experimental details. The un-coated PP-MgO:SLN crystal was operated near the damage threshold since damage did not occur at all operating locations on the crystal.

In spite of the damage, important qualitative data were obtained from the first set of OPO experiments: the OPO did reach threshold, preliminary phase-matching data were acquired and OPO oscillation was observed at the lowest crystal temperature tested (30 °C). Reaching threshold was significant because it proved that crystals produced by top-seeded solution growth were uniform enough to be used in nonlinear frequency conversion devices. The phase-matching data were significant because there was no prior refractive-index data on which to base the OPO design. Since the Sellmeier equation for MgO:SLN was not known, the Sellmeier equations for CLN [19] were used as a starting point in predicting the OPO operating wavelengths. Because a sample of PPLN producing a similar range of signal wavelengths was available, we concluded that the existing OPO mirrors could be used to compare the performance of PP-MgO:SLN and PPLN once the surface-damage problem was solved. OPO oscillation at 30 °C was significant because a similar PPLN OPO could not operate at crystal temperatures below 100 °C. Thus the improved resistance to photorefractive damage of MgO:SLN was qualitatively confirmed at the 1% MgO doping level.

A second, more detailed set of OPO experiments was carried out on coated crystals. To decrease the susceptibility to surface damage, the PP-MgO:SLN crystal was re-polished, and an AR coating was applied, both at Deltronic Crystal Industries. The coating was expected to lower the oscillation threshold by eliminating the Fresnel reflection loss, while also raising the surface-damage threshold. The control sample of PPLN was included in the polish and coating runs. Both samples had the same length after polishing.

The re-polished and coated crystals were inserted into the OPO cavity (output coupling 10%, average pump power 800 mW, peak pump power 3.2 kW). The expected increase in resistance to surface damage was observed; no such damage was seen on either crystal under any OPO operating conditions. Figure 4 shows the tuning data for the PP-MgO:SLN crystal as a function of grating period for different temperatures. The OPO was tuned by translating the crystal transversely across the beam so that the pump interacted with the different grating sections [17]. For a given QPM period, the OPO wavelengths for PP-MgO:SLN were identical to those calculated for PPLN with a slightly shorter (∼ 0.7 µm) QPM period. The OPO would not operate on the 28.2-µm and 28.5-µm gratings at 50 °C, or the 28.2-µm grating at 100 °C. This failure to oscillate can be attributed to the limited bandwidth of the OPO mirrors and crystal AR coatings below 1420 nm, and possibly also to strong absorption at idler wavelengths greater than 4.2 µm [17].

Figure 5 shows the OPO output power as a function of the input pump power. For this measurement, the temperature of the crystals was 150 °C. The PPLN crystal, with a QPM period of 29.0 µm, produced a signal wavelength of 1503 nm. The PP-MgO:SLN was positioned to operate on the 29.7-µm grating period to match the signal wavelength of the PPLN. Both crystals had the same oscillation threshold, but PP-MgO:SLN had a slightly lower slope efficiency.

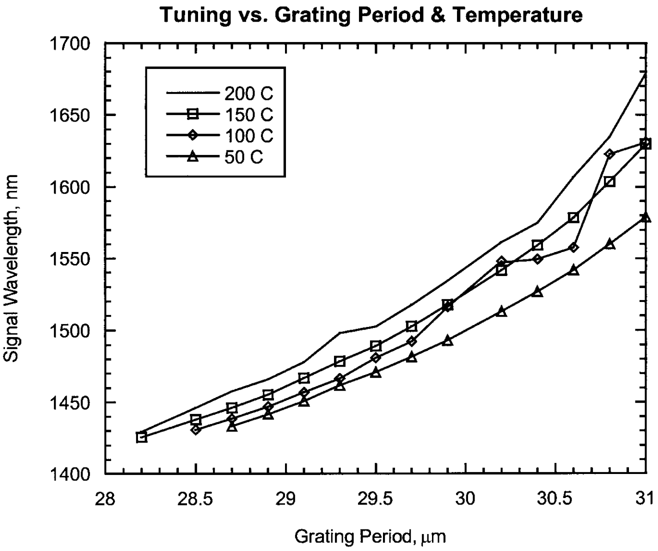

Fig. 4. Signal wavelength vs. QPM period and temperature for PP-MgO:SLN with 1064-nm pump. The scatter in the data at 100°C is probably attributable to experimental error

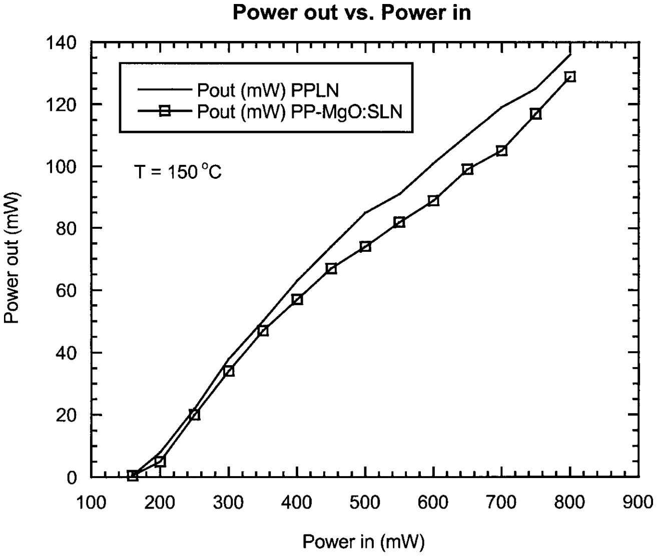

Fig. 5. Power out vs. power in for OPOs using PPLN and PP-MgO:SLN. PPLN: QPM period =29.0 μm, signal wavelength =1503 nm. PP-MgO:SLN: QPM period = 29.7 μm, signal wavelength = 1503 nm

Figure 6 shows the OPO signal power out as a function of temperature. For the conditions of this experiment, OPO operation of both crystals was un-affected at temperatures down to room temperature. It has previously been shown that the photorefractive damage (caused by parasitic radiation at the second harmonic of the pump laser) occurring at low-temperature operation can lead to excess loss which terminates OPO operation. This damage does not occur if the pump power is below a certain threshold value [3, 20]. For congruent un-doped LN, this threshold is believed to correspond to an average second-harmonic intensity of 100 W/cm^2 [21]. For example, in a previous experiment using a similar set-up with PPLN, the OPO was inoperable because of photorefraction below ~ 100 °C with 1 W of average pump power, but operable without degradation down to room temperature with 0.1 W of average pump power [3].

The reason for the lower slope efficiency (in Fig. 5) and lower output power (in Fig. 6) for PP-MgO:SLN is not known. Possible contributing factors include greater material inhomogeneity, and poorer periodic poling quantity, of the first-generation PP-MgO:SLN material in comparison with PPLN which has benefited from years of development work.

The temperature dependence of photorefractive damage can be complicated in an OPO because both the power of the parasitic green radiation which initiates the damage, and the properties of the material, are functions of temperature. For instance, in a separate OPO experiment photorefraction was observed at ~ 100 °C while it was not observed at both higher and lower temperatures, due to increased conductivity at higher temperatures and decreased green generation at lower temperatures [22].

Although no degradation of OPO power was seen in this experiment, evidence of photorefraction was observed visually in the form of fanning and scatter in the visible beams that are produced by extraneous nonlinear interactions during OPO operation. For PPLN, these effects were observed at temperatures below 100 °C, while for PP-MgO:SLN they were observed only at temperatures below 35 °C and even then the effects were less pronounced than they were in PPLN.

Figure 7 shows the temperature dependence of the OPO signal wavelength for both crystals over the range of temperatures covered by the photorefraction tests. To ensure the best possible comparison between the two materials, the QPM periods were chosen so that the operating wavelength would be approximately the same for both materials at each temperature. Because the OPO mirrors and crystal AR coatings have a relatively flat spectral response over the range of observed signal wavelengths (1475 nm to 1520 nm), the cavity losses were relatively independent of wavelength. Because the product of signal and idler frequencies changes by only 4% over this range of signal wavelengths, the OPO gain was

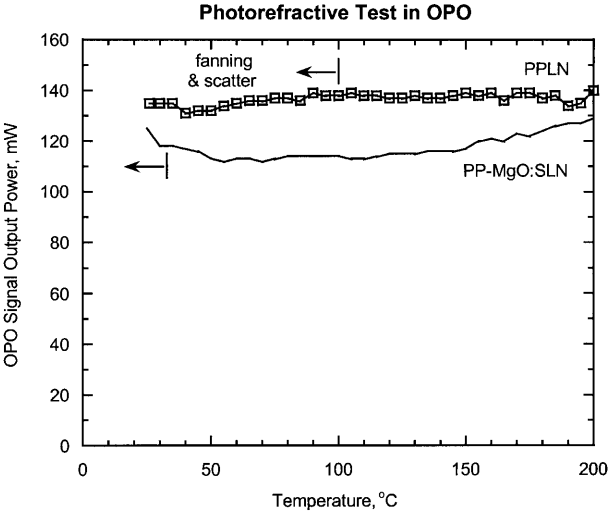

Fig. 6. OPO signal power vs. temperature. The PPLN and PP-MgO:SLN crystals both operated without degradation of OPO power down to room temperature, but there was an indication of photorefraction in the visible beams at lower temperatures

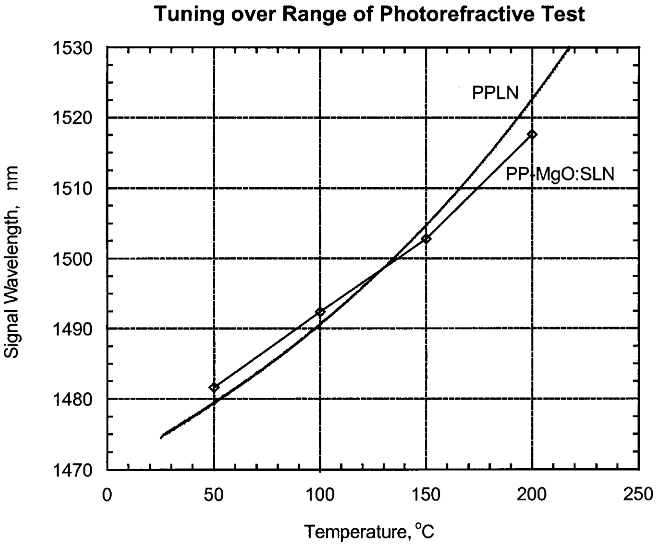

Fig. 7. OPO signal wavelength vs. temperature over the range covered by the photorefractive test shown in Fig. 6. The QPM periods were 29.0 μm and 29.7 μm for PPLN and PP-MgO:SLN, respectively

relatively constant. Therefore, the wavelength dependence of the OPO oscillation threshold can be ignored when analyzing the data shown in Fig. 6.

The OPO experiments compare the relative merits of two materials (PP-MgO:SLN and PPLN) under practical device operating conditions. The relative merit of a third material (PP-MgO:CLN) could not be studied because that material was not available. If this material were available in suitably sized pieces, OPO studies could be carried out to determine its usefulness relative to the two materials studied in this work.

In separate work, the level of GRIIRA in 1% MgO:SLN (grown at Deltronic) was measured using photothermal common-path interferometry [5, 6] and compared with the level of GRIIRA in the baseline material, CLN (produced at Crystal Technology). Neither sample was periodically poled. At a 514-nm pump power density of $1.3\,\mathrm{kW/cm^2}$, the additional steady-state infrared absorption (at a wavelength of 1064 nm) was smaller by more than a factor of 20 in MgO:SLN than it was in CLN. The index change due to photorefraction was also reduced by more than a factor of 20, as measured using the same technique. These results suggest that PP-MgO:SLN will have much greater resistance to the damage caused by GRIIRA than PPLN.

5 Summary and conclusions

In summary, magnesium-oxide-doped stoichiometric lithium niobate has been produced using the technique of top-seeded solution growth from a lithium-rich melt. Optical tests, performed with a combination of argon-ion laser lines, have confirmed a previously published result (at 532 nm) that this material has superior resistance to photorefractive damage. This material has been shown, for the first time, to be amenable to periodic poling. Optical parametric oscillator tests have shown that this material maintains the advantages of periodically poled, congruent, un-doped lithium niobate while showing no evidence of photorefractive damage under typical operating conditions. Operating wavelengths as a function of quasi-phase-matching period and temperature have been measured for the optical parametric oscillator, providing useful new information about refractive-index dispersion in this material. This work establishes periodically poled, magnesium-oxide doped stoichiometric lithium niobate as a viable material for nonlinear optics.

Acknowledgements. We are grateful to Alexei Alexandrovski of Stanford University for communicating results of his GRIIRA measurements to us prior to publication. Portions of the work at Gemfire were supported by the U.S. Air Force and the Ballistic Missile Defense Organization. Work at Lightwave Electronics was supported by the U.S. Air Force.

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