Single-mode rib waveguides in (Yb,Nb):$RbTiOPO_4$ by reactive ion etching

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We present the fabrication of single-mode rib waveguides in (Yb,Nb):RbTiOPO4 ((Yb,Nb):RTP) by reactive ion etching (RIE) with a combination of SF6 and Ar gases. The influence of the gas pressure, RF power and gas ratio on the etch rate and surface quality was studied to optimize the etching of RTP. Optimized parameters were used to fabricate rib waveguides in a (Yb,Nb):RTP film, grown by liquid phase epitaxy, with an etch rate of 10 nm/min. Channel waveguide propagation was demonstrated for the first time in an RIE etched (Yb,Nb):RTP rib structure.

1. Introduction

Ytterbium (Yb^{3+}) doped gain media are useful materials for the realization of efficient power-scalable lasers [1]. They operate near the 1 μm spectral regime and have the advantages of a broad absorption band making them suitable for high-power diode pumping, low thermal load due to a small quantum defect and negligible losses due to parasitic processes such as up-conversion and excited state absorption. Recently, laser operation has been demonstrated in Yb^{3+}-doped $RbTiOPO_4$ (RTP) crystals [2]. RTP belongs to the $KTiOPO_4$ (KTP) class of orthorhombic crystals and is therefore of interest for nonlinear applications. When doped with Yb^{3+}, RTP has a very broad emission bandwidth [3] making it a good candidate for the generation of ultrashort pulses, with pulses as short as 155 fs being demonstrated to date [4].

(Yb,Nb):RTP gain media can be grown in a waveguide geometry by liquid phase epitaxy (LPE) [5] and planar waveguides with losses lower than 1 dB/cm have been demonstrated. Fabrication of channel waveguides based on this thin-film growth technology is motivated by noting recent work on ion-exchanged glass waveguides [6], showing that Yb^{3+}-doped channel waveguides are of interest as compact ultrafast sources with multi-GHz repetition rates, and consequently have potential application in areas such as optical sampling [7], metrology [8] and biological imaging [9]. The non-linear properties of the RTP crystal could also be utilized for fabricating compact self-frequencydoubled [3, 10] integrated waveguide sources, which would have enhanced performance in a low-loss channel waveguide geometry.

Structuring of RTP has been demonstrated using femtosecond laser ablation [11] and wet chemical etching [12]. Reactive ion etching (RIE) is a commonly used method for etching materials in the semiconductor industry [13], however there is a lack of literature on the plasma-based micromachining of RTP and to date there haven't been any demonstrations of channel waveguide structures in RTP.

Chlorine-rich gases have been extensively used for etching materials by RIE, but chlorine-based plasma is toxic and can be damaging to the chamber. Fluorine based gases are more environmental friendly and RIE of other interesting candidates for ultrafast waveguide lasers, such as sapphire [14] and KYW [15], have both given promising results with fluorine-rich chemistry. In this paper we explore the RIE of RTP in a fluorine-rich environment with the view of fabricating channel (Yb,Nb):RTP waveguide lasers. We have performed an extensive study of the etching characteristics of RTP by RIE with SF_6 and Ar gases by varying the RF power, gas pressure and gas ratio. On correlating the etch rates and surface roughness of the etched regions with different conditions we reached the optimum conditions for etching RTP. The optimum parameters were used to fabricate channel waveguides in a LPE-grown (Yb,Nb):RTP film with an etch rate of 10 nm/min. The channels had a measured surface roughness of 8 nm and a side-wall angle of 63º. Single-mode propagation was demonstrated in these channels with mode–radii of 8.2 µm by 4.1 µm.

2. Growth of single crystals for substrates and liquid phase epitaxy growth of films

The RTP crystals used as substrates for the etching experiments were grown in a tubular furnace [16] using the top-seeded solution growth method (TSSG) and super-saturation of the solution was obtained by slow cooling [17]. We used an RTP seed parallel to the c crystallographic direction, since this orientation is one of the best to grow high-quality RTP crystals and from these crystals it is possible to obtain substrates with suitable dimensions in the ***ab*** plane. In fact, this plane has interest for non-critical phase matching type-II SHG [3] for fundamental radiation around 1µm, which is close to the emission wavelength of Yb^{3+}. Moreover, the ***ab*** plane is perpendicular to the orientation of the ferroelectric domains, which makes it possible to obtain a periodic domain inversion, producing quasi phase matching [18]. All the substrates were cut perpendicular to the c-crystallographic axis and polished.

LPE growth of a (Yb,Nb):RTP thin film was carried out in a furnace where a zone of uniform temperature suitable for epitaxial growth was obtained. The composition of the solution used for epitaxial growth was Rb_2O – P_2O_5 – (TiO_2 + Nb_2O_5 + Yb_2O_3) – WO_3 with concentration of 43.90-23.6-22.50-10.00 (mol%) respectively. TiO_2 was partially substituted by Nb_2O_5 and Yb_2O_3 up to concentrations of 2 and 6 (mol%), respectively with Nb_2O_5 co-doping allowing higher concentrations of Yb^{3+} to be obtained [19]. After the homogenization of the solution, the substrates were slowly introduced into the furnace to avoid possible cracks due to thermal stress. The substrates were then dipped into the solution and kept at 1K above the saturation temperature for 5 minutes to dissolve the outer part of the crystal. To begin the epitaxial growth, the temperature of the solution was decreased to 9K below the saturation temperature in order to create super-saturation in the solution, which is the driving force behind the crystal growth. The substrates were stirred at 60 rpm and maintained under these conditions for 6 hours. The obtained epitaxial layers were morphologically studied using a Sensofar PLµ 2300 interferometric microscope in order to check if they were free of defects and to measure the film thickness, which was initially 70 µm and then polished to a uniform thickness of 6µm. The dimensions of the substrate were 12x8x2 mm. An electron probe micro-analyzer was used to measure the concentration of Yb^{3+} in the epitaxial layer and it was found to be 0.33 at.%, which corresponds to an Yb^{3+} concentration of $2x10^{20}$ cm^{-1}. The refractive index contrast in TM polarization (E//c-axis) was measured to be $4x10^{-3}$ at a wavelength of 972 nm (from a Ti:sapphire laser) by m-line measurements.

3. Optimisation of the RIE of RTP

The etching of RTP by RIE was optimised to reduce the RMS surface roughness in order to fabricate low-loss waveguides. A 300 nm layer of Cr was deposited by e-beam evaporation after which a 1.3µm layer of S1813 resist was photolithography patterned to give waveguide features of widths ranging from 1 µm to 10 µm. The Cr layer was then wet etched and the resist was removed by solvent cleaning. The hard-masked RTP substrates were then etched in an RIE chamber with SF_6 and Ar gases where the pattern of the Cr was transferred on the RTP substrate, following which the Cr mask was chemically removed (the stages involved are shown in figure 1). RIE was carried out in an OPT Plasmalab 80 plus RIE system (Oxford Instruments) with an RF frequency of 13.56 MHz.

The RF power, the gas pressure and the gas ratio were the three parameters which were varied during RIE. An RF power of 250 W, a pressure of 50 mTorr , a total gas flow rate of 20 sccm and a gas ratio of 90:10::SF_6:Ar were chosen as starting parameters. The temperature was kept fixed at 20°C for all the experiments. SF_6 was selected because this gas provides more free fluorine radicals [20] when compared to the other available fluoride gas, CHF_3. In contrast, Ar is an inert gas and is predominantly used to bombard the surface and hence make the etching process more physical. A 90% SF_6 gas mixture was used initially in order to have a predominantly chemical process. These parameters were then varied systematically to study their effect on etch rate and surface roughness. The measured substrate RMS surface roughness before etching varied from 2 nm up to 5 nm from sample to sample; hence the change in the RMS surface roughness (after etching-before etching) has been presented in figure 2.

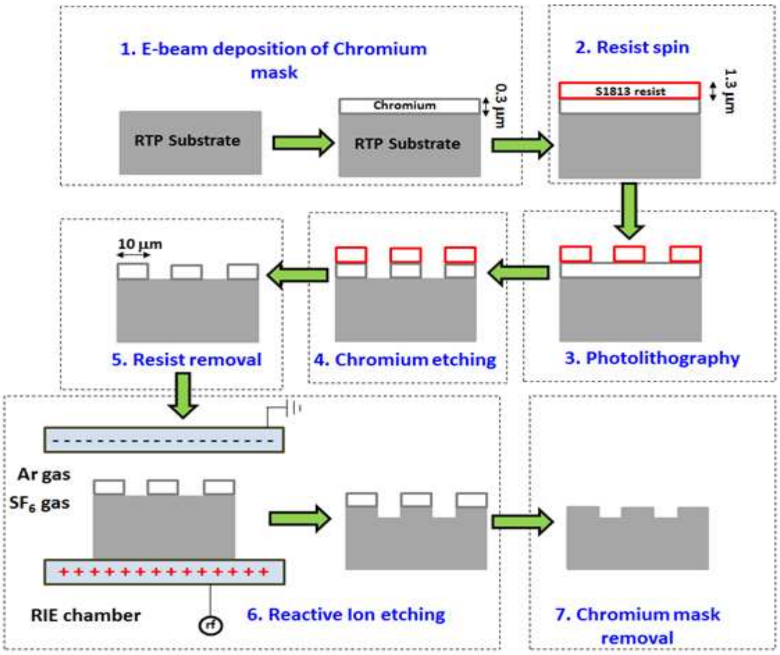

Figure 1. Different steps involved in the RIE of RTP.

Keeping all the other parameters fixed and an etch time of 40 minutes, the RF power was varied and the results obtained for etch rates of RTP and Cr, as well as the RMS surface roughness of RTP are presented in figure 2.a. As expected, the etch rate increases linearly with power, and an etch rate as high as 13 nm/min is obtained for an RF power of 350 W. The surface roughness also increases with the power and a surface roughness of 3.4 nm is obtained for 350 W RF power. The power was not increased beyond 350 W as the value of surface roughness becomes too high beyond this point.

A good balance was achieved between surface roughness and etch rate for a power of 250 W and hence the power was fixed at 250 W and the SF_6:Ar ratio was fixed to 90:10 for the next set of experiments. The chamber pressure was then varied and the results obtained for etch rates and surface roughness are presented in figure 2.b. The etch rate decreased with pressure. This is because of the increase in scattering with pressure and hence a decrease in the mean free path for the radicals [21] and a loss of kinetic energy. It was found that a pressure of 50 mTorr resulted in an etch rate of 8 nm/min and a surface roughness of 1.6 nm. Thus, this pressure was fixed for the study of etch rate and surface quality dependence on the gas ratio.

The results obtained for etch rates and surface roughness against gas ratio are presented in figure 2.c. At low SF_6 concentrations, the etch rate is very high (16 nm/min) and so is the surface roughness (3.4nm). This is a predominantly physical process and a lot of the material is sputtered by the Ar^+ ions. At very high SF_6 concentrations, the etch rate decreases, but the surface roughness also increases slightly. The optimum operation point for gas ratio (SF_6:Ar) is in the range of 50:50 to 70:30, where the etch rate is about 8 nm/min and the surface roughness is less than 1 nm.

The DC bias of the RIE chamber is process parameter dependent. For each measurement described above, the DC bias was measured and the etch rate of RTP and Cr versus the DC bias are plotted in figure 2.d. The graph shows that the etch rate increases with increasing DC bias, which is as expected [22]. It is important to note that all the data points do not have the same conditions, however the linear behaviour of the etch rate shows consistency in the plasma condition against the gas ratio, pressure and RF power. As seen from figures 2a-2c , an achieved etch rate of 8-10 nm/min gives a good balance between etch rate and surface roughness, this corresponds to a DC bias of around -300V as seen from figure 2.d. The selectivity (ratio of etch rates of RTP to chromium) was found to be greater than 5 for almost all conditions. This allows the use of thin Cr masks for deep etching of RTP.

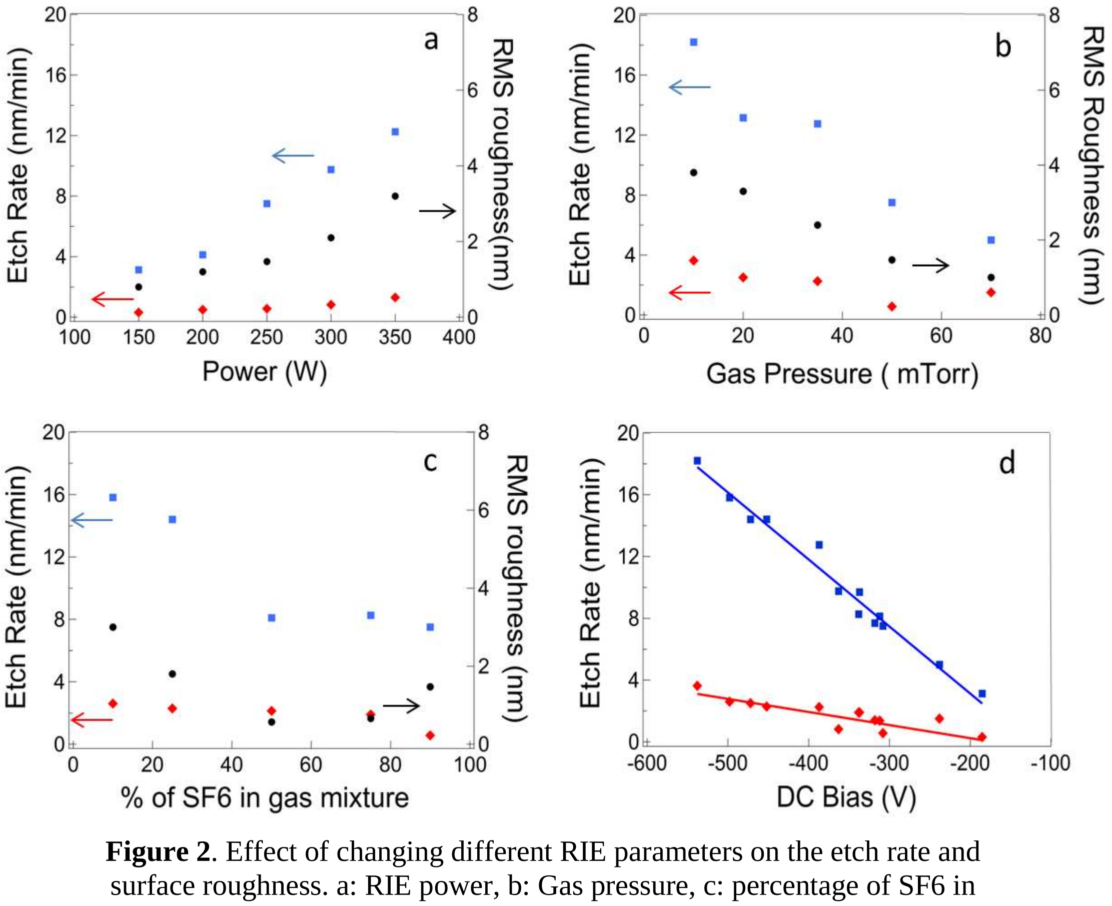

Figure 2. Effect of changing different RIE parameters on the etch rate and surface roughness. a: RIE power, b: Gas pressure, c: percentage of SF6 in gas mixture, d: DC bias. Blue: etch rate of RTP, red: etch rate of Cr, black: RMS surface roughness of the etched surface.

4. Channel waveguides in (Yb,Nb):RTP

The (Yb,Nb): RTP thin-film described in section 2 was etched for 135 minutes with RIE power of 250 W, gas pressure of 40 mTorr and gas flow rates of 10 sccm for both SF_6 and Ar . The pressure was reduced to 40 mTorr from 50 mTorr to increase the etch rate without compromising the surface roughness. This sample was then end-polished to a length of 7.5 mm, cleaned in an ultrasonic bath with acetone, isopropanol and DI water, blow-dried with N_2, and finally dehydrated in an oven at 90ºC for 10 minutes. The mask was chemically removed, after which the sample was plasma-ashed in an RIE chamber for 20 minutes with RIE power of 200 W, pressure of 50 mTorr and O_2 flow rate of 10 sccm, which removed any organic impurities present on the substrate. The etch depth was measured to be 1.4±0.1 μm, giving an etch rate of 10 nm/min. This etch depth was chosen to support a fundamental mode near 1 μm. The surface roughness of the film before etching was 3±0.5 nm, and after etching it was measured to be 8±0.5 nm. The fact that it is slightly higher when compared to the expected values from figure 2 could be due to the longer etch time for this process combined with re-deposition of the mask on the un-etched regions. Figure 3 shows the SEM images of the etched sample. For a waveguide with a top width of 9.8 μm, the width at the bottom was 11.2 μm giving a sidewall angle of 63º.

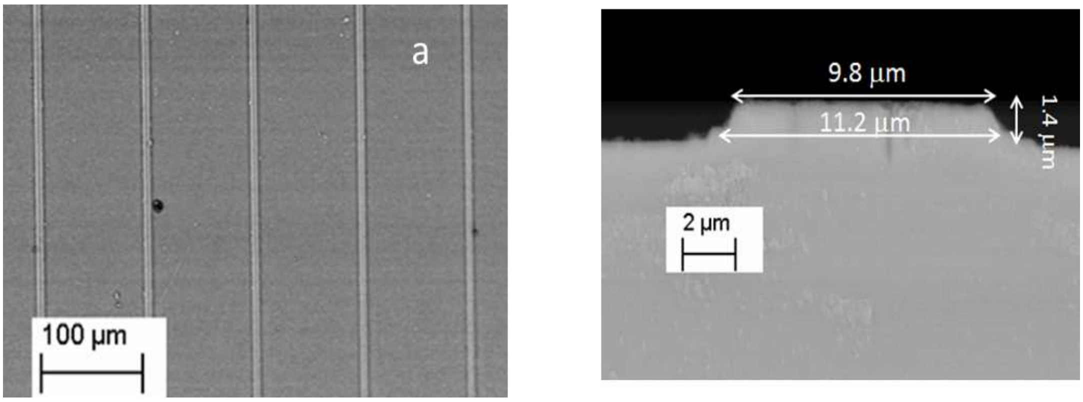

Figure 3. SEM images of the (Yb,Nb):RTP samples etched with RIE power of 250W, SF_6=10 sccm, Ar=10 sccm and pressure= 40mTorr. a: top view b: cross-section of waveguide with top width of 9.8 μm

The surface topography of the planar region and the ribs measured by a non-contact profiler (Zescope by Zemetrics) is shown in figure 4. It can be seen from figure 4.a. that the surface quality, barring a few spikes (possibly due to the re-deposition of the mask), is very good.

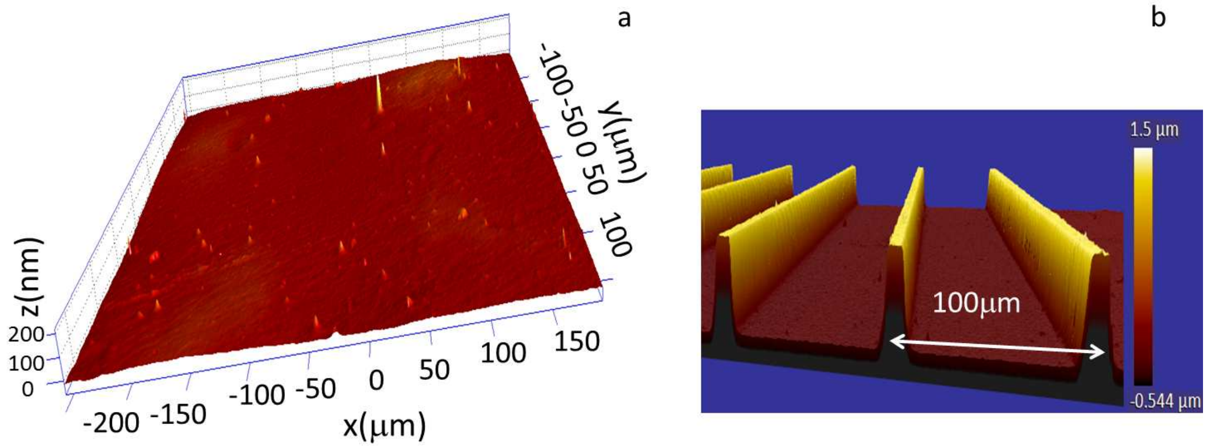

Figure 4. Surface topography of an etched planar region (a) and a region with etched ribs (b) measured by non-contact profiler (Zescope by Zemetrics).

Optical characterization was carried out by end-fire coupling a fiber-coupled single-mode laser diode from 3S photonics with a maximum power of 750 mW and a grating stabilized wavelength of 980.6 nm. The collimated pump was passed through a λ/2 plate to control the polarization. This was coupled into the waveguide by a 16x objective giving a measured waist radius of 3.2 µm. The output from the waveguide was collected by a 6x objective and imaged on a camera to measure the mode profile or was incident on a power meter for transmission measurements. The losses were estimated by comparing the transmitted light from the channel waveguide with the incident light and accounting for Fresnel reflections, transmission of the objectives etc.

Transmission was measured at various powers and losses were estimated to be <3.5 dB/cm for the E//c crystallographic axis (TM). This calculation assumes 50% launch efficiency due to the imperfect match between the launch mode size and shape compared to the propagation mode but it may be considerably worse and hence the losses may be significantly lower.

On rotating the polarization, the losses increased to >10 dB/cm for TE polarization. This is consistent with the fact that the index contrast is very low for this polarization. It should be noted that in this configuration, the spectroscopy of (Yb,Nb):RTP means that TM is the preferred polarization for lasing [2]. The waveguide structure is designed to be single mode at 981 nm and the simulated and measured TM profile is shown in figure 5. The $1/e^2$ mode radii were measured to be 8.2 µm and 4 µm in the x and y directions respectively. This is in good correlation with the simulated values of 7.6 µm and 3.4 µm along the x and y directions respectively (simulated by a commercially available software OlympIOs). The slight distortion seen (marked in red in figure 5.b.) could be due to scattering.

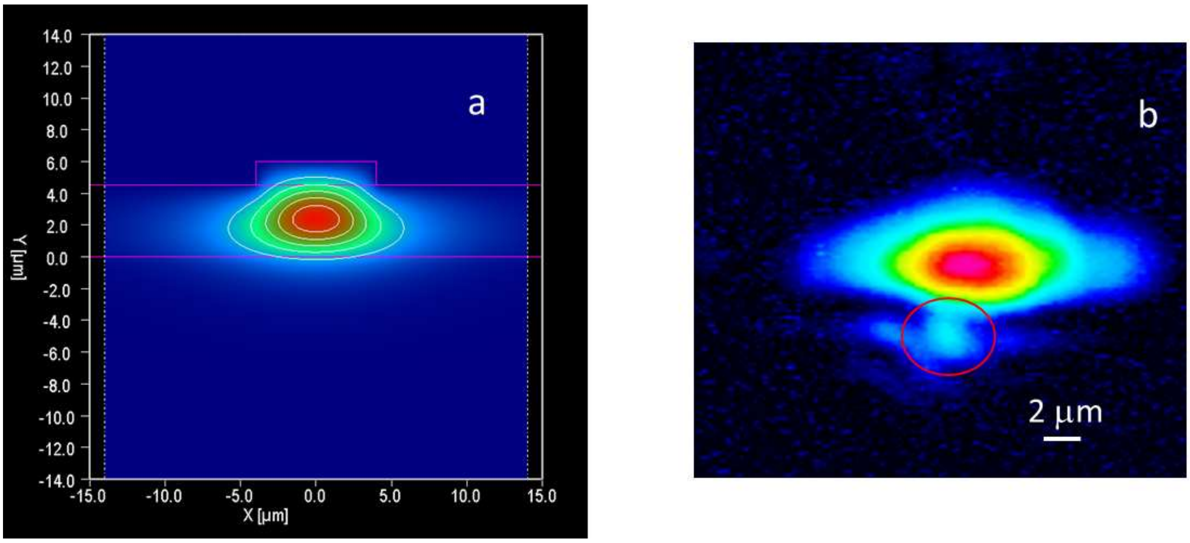

Figure 5. a: Simulation of the mode profile in an 8 µm wide (Yb,Nb):RTP rib waveguide at a wavelength of 0.98 µm. b: Measured near-field mode profile for the RIE etched waveguide.

5. Conclusions

Optimization of the reactive ion etching of RTP was systematically carried out by varying the process parameters: RIE power, SF_6 and Ar gas ratios and gas pressure. This led to optimized parameters; RIE power of 250 W, gas pressure of 40 mTorr, 50:50 gas ratio and total gas flow rate of 20 sccm. These parameters were used to fabricate single-mode rib waveguides in (Yb,Nb):$RbTiOPO_4$ for the first time to our knowledge. Single-mode waveguides with mode radii of 8.2 µm x 4.1 µm were realized with losses estimated to be <3.5 dB/cm by transmission measurements. Such waveguides could have promising applications in ultrafast and self-frequency doubling lasers.

6. Acknowledgments

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