Photorefractive Damage in congruent LiNbO$_3$.
Part I. Zinc doped Lithium Niobate Crystals

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Abstract. The experimental measurements of the photorefractive damage in a series of zinc doped congruent lithium niobate are reported and discussed. The first experimental technique used is based on the measurement with time of the photoinduced distortion of an HeNe beam spot at 632.8 nm. The second method is based on the direct measurement of the photoinduced birefringence variation with time, using a powerful laser beam at 514.5 nm as gating radiation, and a laser weak beam at 632.8 nm as probe. The dependences of the photorefractivity and of the photosensitivity on the power and dopant concentration have been investigated. The photorefractive damage is highlighted by the electro-optic behaviour with the zinc concentration and the defect structure of the doped crystals. We conclude that doping above a second threshold concentration with divalent Zn ions leads to a significant decrease of the photorefraction with respect to pure congruent crystals. In this concentration range, LiNbO$_3$:Zn are strongly photorefractive damage resistant and thus present an interesting alternative for modulating devices to the stoichiometric lithium niobate. Link to the high electro-optic coefficients, these crystals are very suitable for Q-switching applications.

1. Introduction
Lithium niobate (LN) crystals have good optical quality and uniformity, an excellent electro-optic, piezoelectric, acoustic and nonlinear optical properties. Generally, due to their physical properties but also because of their growth facilities, LN crystals with congruent composition, corresponding to a ratio $[\text{Li}]/[\text{Li}+\text{Nb}]=48.6\%$, are generally used in a lot of optical and acoustic devices. Nevertheless, the main drawback of congruent lithium niobate for successful applications in devices is its relatively low optical damage resistance compared to other competing oxide crystals such as LiTaO$_3$ (LT) or BaB$_2$O$_4$ (BBO) [1,2]. The optical damage corresponds to local modifications of the refractive indices of the medium induced by light beam [1]. The main process in LN crystals is the photorefractive damage due to the presence of space charge field induced by the optical beam, which generate mobile charge carriers in the bright region and migrate toward the dark zones. Consequently, induced via the electro-optic effect, the space charge field thus creates modifications of refractive indices. For general considerations, another origin of the optical damage could be due to thermal effects, such as the thermo-optic effect. In this effect, the power density of the laser beam induces a local heating in the...
crystal that changes its refractive indices. Nevertheless, in LN crystal, for relatively high power densities of the beam, these effects are negligible compared to the photorefractive effect [3].

In order to reduce the photorefractive damage in LN or to improve some non-linear optical properties, different approaches have been developed. One of them consists of decreasing the number of intrinsic defects by growing LN crystals with the ratio \( R = \frac{[\text{Li}]}{[\text{Nb}]} \) closer to the one corresponding to the stoichiometric composition (\( R = 1 \) and \( R = 0.946 \) for stoichiometric and congruent compositions, respectively). But stoichiometric crystals cannot be grown by conventional Czochralski technique even in melt with ratio \( R \) far above one. The growing process is delicate and stoichiometric lithium niobate cannot be yet produce in large quantities and constant qualities as necessary for applications. The second approach consists in doping congruent lithium niobate crystals by appropriate doping ions like divalent ions such as \( \text{Mg}^{2+} \) [4,5] and \( \text{Zn}^{2+} \) [6] or trivalent ions such as \( \text{Sc}^{3+} \) and \( \text{In}^{3+} \), which are known, for specific concentrations, to improve the optical damage resistance of LN crystals. Moreover, it has been shown that doping with hafnium also reduces the optical damage in congruent LN crystals [7]. Significant reduction of optical damage was observed at doping concentrations about 5.5 mol.\% \( \text{MgO} \) [4,5], 7 mol.\% \( \text{ZnO} \) [6], 1.5 mol.\% \( \text{Sc}_2\text{O}_3 \) [8], 1.5 mol.\% \( \text{In}_2\text{O}_3 \) [9] and 4 mol.\% \( \text{HfO}_2 \) [7].

The purpose of this paper is to investigate the optical damage in four congruent crystals doped with increasing \( \text{ZnO} \) concentrations. The techniques used for these characterizations were already described in previous papers [10,11]. We present the behavior of the photoinduced birefringence change at saturation and the photoconductivity as a function of the power densities of an Argon pump beam for the two sets of crystals. In order to clarify our analysis, the results will be confronted with those obtained in electro-optical characterizations made on the same crystal samples.

It is to be of note that the optical damage investigations in congruent lithium niobate are presented in two publications. The present part is dedicated to the photorefractive behavior of zinc doped congruent LN, which is analyzed after the presentation of the two methods used for the characterizations. Then, the second part follows with the presentation of the photorefractive damage of a series of magnesium doped congruent LN done within the same experimental processes and which finishes with a general discussion on the behavior and its comparison of these two doped LN families. Both publications were written to be self-consistent and to be read independently.

2. Materials and Methods

2.1. Crystal presentation

We have performed photorefractive measurements in four congruent zinc doped crystals. In order to discard the influence of other involuntary kinds of impurity, crystals have been prepared in the same conditions. This set of crystals is made up of LN crystals doped with different concentrations of zinc and provided by T. Volk [6]. These crystals were grown using the Czochralski method from congruent melt to which Zn was added in its oxide form. The concentrations of doping ions in the crystals are listed in Table 1; they are based on the concentration in the melt using the empirical equation proposed by Schlarb et al. [13].

The optical quality of all crystals was checked by an optical transverse mapping, which shown in the visible range a good optical uniformity without polishing or growth striations, respectively at the surface or inside crystals. The nonuniformity of the birefringence along the axis does not exceed 10-5 cm-1. For a suitable comparison, Y-cut plates with the same thickness equal to 0.68±0.02 mm were especially prepared for the laser-induced birefringence measurements presented in this paper.

To determine the role of Zn and LN structural defects in the discussion, we have introduced experimental results obtained with a pure congruent LN crystal; unfortunately, this crystal was not grown in same conditions than the Zn series and additional effects due to impurities (as iron at various valence states) have to be considered.
### Table 1. Labels and concentrations of Zinc doped LN crystals

| Crystal | Me-oxide in the crystal (mol.%) |
|---------|---------------------------------|
| Zn-1    | 2.90                            |
| Zn-2    | 3.78                            |
| Zn-3    | 6.40                            |
| Zn-4    | 7.75                            |

2.2. Experimental methods

Photorefractive damage measurements were carried out by means of the two methods presented in details in Ref. [10]. We just summarize here, the fundamentals of both methods necessary for a good understanding of the relevant parameters measured in the experimental work to characterize the photorefraction of the samples.

The first method consists of the direct measurements of the self-defocussing of a laser beam photo-induced in the crystal. This method is based on the implementation of the pseudo Z-scan method where the sample is fixed at the waist position of the laser beam and measurements of transmitted intensity are done through the sample and a pinhole.

The curves of variation of transmission as a function of time can be fitted by a first-order exponential function:

\[
T(t)(\%) = \frac{I}{I_0} = T_{\text{min}} + \Delta T \exp(-t/\tau) \tag{1}
\]

where \(T_{\text{min}}\) is the minimal transmission at saturation, \(\Delta T = 100 - T_{\text{min}}\), \(I_0\) and \(I\), the transmitted beam intensities at \(t=0\) and at a time corresponding to the saturation of the scattering respectively and \(\tau\) is the dielectric relaxation time which is linked to the dielectric constant and photoconductivity by:

\[
\tau = \frac{\varepsilon_0 \varepsilon_{33}}{\sigma_{\text{ph}}} \tag{2}
\]

In this method the relevant characteristics are the variation \(\Delta T\) of the transmitted laser beam and \(\sigma_{\text{ph}}\).

The second method was implemented for the direct determination of the photoinduced birefringence change \(\delta \Delta n\), using a very sensitive set-up, based on a modified FDEOM method detailed in Ref. [14] and presented in its adaptation form for photorefractive measurements in Ref. [10]. Within this method, the optical damage is measured by monitoring the change in the magnitude of the induced birefringence as a function of irradiation time. Two aligned and concentric laser beams with different wavelengths are used for the pump and for the probe. The focused pump cw laser beam induces in the crystal an internal space charge electric field due to the displacement of carriers between the bright and the dark zone. This field involves a modification of the refractive indices via the electro-optic effect and as a consequence, the output phases of both optical beams are modified. The induced change of the probe beam phase is directly monitored on an oscilloscope screen, which yields to the value of the light-induced birefringence changes \(\delta \Delta n\), via the well-known equation

\[
\delta \Delta n = \frac{\delta \Gamma \lambda}{2\pi L} \tag{3}
\]

where \(\delta \Gamma\) is the induced phase shift, \(\lambda\) is the wavelength of the probe beam and \(L\) is the width of the sample. The changes were monitored as a function of irradiation until saturation. The curves obtained within this method can be fitted with a first order exponential time response function given by
\[ \delta \Delta n(t) = \delta \Delta n_s [1 - \exp(-t/\tau)] \]  

(4)

where \( \tau \) is the characteristic time of the PR effect (as in Eqs. 1 and 2) and \( \delta \Delta n_s \) the saturated value of the birefringence change. In this method the relevant characteristics are \( \delta \Delta n_s \) and \( \sigma_{ph} \).

3. Results and discussion

3.1. Self-defocusing measurements

We have measured the self-defocussing of an He-Ne spot beam at a wavelength of 633nm passing through the crystal and a power density at the focal plane equal to 475 W/cm². For each crystal, we have recorded as a function of the illuminated time, the two beam intensities \( I \) and \( I_0 \), to determine the characteristic \( T(t) \) (Eqs. 1). From the fits of the \( T(t) \), we deduce the maximum light-induced scattering \( \Delta T \) corresponding to the saturation obtained after few minutes in the change of \( T(t) \), \( \Delta T(t) \) and finally, via Eq. 2, the value of the photoconductivity \( \sigma_{ph} \).

We have plotted in Fig 1, experimental results obtained as a function of Zn concentration in crystals. We can see, in Fig. 1 that the light-induced scattering \( \Delta T \) decreases monotonically with increasing Zn contents. No concentration threshold seems to appear in this concentration range. The behaviour of the photoconductivity is opposite among that of \( \Delta T \) corresponding to a monotonous increase.

![Figure 1](image)

**Figure 1.** Variations of the transmittance \( \Delta T \) and photoconductivity \( \sigma_{ph} \) as a function of Zn concentration in LN:Zn crystals for a laser beam intensity equal to 475 W/cm² at \( \lambda = \) 633nm.

An interpretation of these behaviors will be suggested below in the discussion with considerations of electro-optic experimental results obtained on these crystals

3.2. Photoinduced birefringence change

We have measured the photoinduced birefringence changes as a function of time, \( \delta \Delta n(t) \) with a focused low-intensity He-Ne laser at a wavelength of 633nm for the probe beam and a focused cw argon laser beam laser oscillating at 514.5nm with a power density of 1.2 kW/cm² for the pump beam. For each crystal we have determined the light-induced phase shift as a function of the illuminated time, \( \delta T(t) \) and we have calculated, using Eq. 3, the value of the maximum birefringence changes at saturation \( \delta \Delta n \), and by Eq. 4, via the dielectric relaxation change, the photoconductivity \( \sigma_{ph} \). At this power density and wavelength for the pump beam, we have report in Fig. 2 the photoinduced
birefringence change at saturation and the photoconductivity versus dopant concentration. In our measurements, $\delta \Delta n$ is the sum of birefringence changes caused by the photorefractive effect and that caused by the thermo-optic effect, both due to the Ar-laser irradiation. Nevertheless when the Ar-laser irradiation is stopped after illumination, no significant birefringence changes occurred even some hours later. That means that the birefringence change due to thermal effects is negligible and that the measured $\delta \Delta n$ in this samples is predominantly induced by the photorefractive effect, which confirm results obtain by Furukawa et al. in pure LN crystals [3]. We can also consider that the relative stability after the end of illumination indicates a weak dark conductivity $\sigma_d$ in all these crystals compared to the photoconductivity $\sigma_{ph}$, which is also confirmed by other authors in pure and doped crystals [2]. Thus, the observed effect is mainly due to the photovoltaic space charge field generated along c-axis. We emphasize that under these experimental conditions, a low intensity probe and high intensity pump focus beams, photorefraction is only due to the bulk photovoltaic effect generated by the pump beam.

Figure 2. Photoinduced birefringence change at saturation and photoconductivity as a function of Zn concentration in LN:Zn crystals for a laser beam intensity equal to 1.2kW/cm$^2$ at $\lambda = 514.5$ nm.

We can see in Fig. 2 that $\delta \Delta n$s have the same behavior versus Zn concentration than the intensity variations, $\Delta T$, measured with the first method and reported in Fig. 1. Moreover, these measurements done with a different method as those presented above confirm the values and the behaviour of the photoconductivity.

The $\delta \Delta n$s dependence on the specific value of the optical damage resistant doping concentration in crystals was earlier connected with the changes of photoconductivity $\sigma_{ph}$ [14]. Doping with an optical damage resistant impurities has the same effect on the optical damage resistance as increasing with the composition ratio R of pure crystal. This dependence between $\Delta \delta n_s$ and $\sigma_{ph}$ with the doping concentration could be also correlated to the amount of intrinsic defects.

4. Discussion

The optical damage resistance of LN crystals doped with specific optical damage-resistant ions has been widely discussed (see by example Ref. [15]). According to the simple photovoltaic model writing by expression [16]

$$\delta \Delta n = r_e \alpha K \frac{I}{(\sigma_{ph} + \sigma_d)}$$

(5)
where $r_{\text{eff}}$ is the effective electro-optic coefficient, $k$ the Glass constant and $\alpha$ the optical absorption coefficient. We can explain a decrease in photoconductivity results in an increase in the birefringence change.

In order to understand the role in the variation of photorefractivity with the amount of optical damage resistant doping ions in lithium niobate, we have considered the photorefractive sensitivity, $\delta S$ like defined by Jermann et al. in Ref. [15]. In ferroelectric crystals, as the main mechanism that controls the electron transport in the absence of an external electrical field is the photovoltaic effect, $\delta S$ is directly connected to the bulk photovoltaic current. The photorefractive sensitivity is considered as a figure of merit for the comparison of the photorefractive behaviour of crystals and is defined as:

$$\delta S = \frac{\Delta n}{I \tau} = \frac{r_{\text{eff}}(j_{\text{ph}}/\varepsilon_0 \varepsilon_r)}{\tau}, \quad (6)$$

where $I$ is the light beam intensity and $j_{\text{ph}}$ is the photovoltaic current density. Fig. 3 shows the variations of $\delta S$ versus Zn ions concentration.

The photosensitivity can be suitably used to determine and discuss the PR properties and their origin regardless to the model. Indeed a large resistance to PR optical damage requires large photoconductivity and low photoinduced birefringence change $\delta \Delta n$ related to the PV current. This current and the photoconductivity have generally a complex dependence with the laser intensity, the composition and the concentration of optical damage resistant ions like shown in Fig. 3.

![Figure 3. Photosensitivity for three power densities of the spot beam as a function of Zn concentration in LN:Zn crystals.](image-url)

We can hence deduce from these curves of $\delta S$ versus Zn dopant concentration, that the highly doped crystal is the most damage resistant.

The characteristic photorefractive time is link to the dielectric constant $\varepsilon_0$ and the relative dielectric constant $\varepsilon_r$, so we can express, from Eq. 6, the photovoltaic current as

$$j_{\text{ph}} = \Delta n \sigma_{\text{ph}}/r_{\text{eff}} \quad (7)$$

To calculate $j_{\text{ph}}$ and to define parameters that occur in the photovoltaic properties of LN crystals, we have also performed measurements of the effective electro-optic (E/O) coefficient involved in the optogeometric configuration corresponding to the $y$-cut samples i.e. $r_{\text{eff}} = r_{33} = (n_0/n_e)r_{13}$. These samples are obtained by the same growing process and in the same conditions that samples used for PR measurements. On the other hand, we have disposed for this part of the study more samples than
for the PR measurements. The experimental method implemented for the E/O measurements, based on a Sénarmont optical arrangement, is the FDEOM method largely detailed in [20]. This method, from which is originate the MFDEOM method [14] used for the photorefractive characterization in this study, offer a great accuracy in the determination of the dc electro-optic coefficients, better than 5%, in the determination of the dc electro-optic coefficients. The dc EO phase retardation in the crystals under test allows the calculation of the coefficients \( r_\text{c} \) [21] and its dependence versus the dopant concentrations, which is reported in Fig. 4. The electro-optic coefficient, \( r_\text{c} \) versus Zn doping concentration, plotted on figure 4, presents a large amplitude of a non monotonous behaviour.

![Figure 4. EO coefficient rc versus Zn concentration in the crystal (The line is just a guide for the eyes).](image)

In this Fig. 4, three concentration ranges may be distinguished. In the low concentration range up to 2-3 mol\% Zn, \( r_\text{c} \) strongly decreases down to a minimum; in an intermediary range up to around 7.5 mol\% Zn, \( r_\text{c} \) abruptly grows reaching a maximum; finally, in high concentration range, \( r_\text{c} \) sharply decreases.

Finally, using these experimental values in the Eq. 7, the bulk photovoltaic current can be calculated and its behaviour is plotted in Fig. 5 in function of Zn concentration.
Figure 5. Photovoltaic current versus Zn concentration in LN:Zn crystals for a laser beam intensity equal to 1.2kW/cm² at $\lambda = 514.5$ nm (The line is just a guide for the eyes).

The bulk photovoltaic current of Zn doped LN samples presents quite the same behaviour that the photoconductivity in spite of the large non-monotonous variation in the electro-optic coefficients with the doping concentration. Thus, these data clearly indicate that the main contribution to the variation of photorefraction comes from variations in $1/\sigma_{ph}$ with the concentration.

Now, we try to correlate the dependencies of the photorefractive and electro-optic properties on Zn concentrations to variations in the defect structure. In literature, the low concentration range, below the first extremum at 2-3 mol% Zn, is often regarded as a specific range in the concentration dependence of some properties such as photoconductivity and photorefraction [22] and the concentration of 7 mol% Zn is usually referred to as a concentration threshold and manifests itself as a second extremum in the concentration dependence of other optical properties of LN:Zn such as refractive indices, IR absorption, phase matching temperature of SHG, etc… [16,17]. An analysis of the influence of Zn doping on electro-optical properties and structure parameters of LN crystals was proposed in Ref [21].

The two proposed models of defect structure in LN doped by divalent ions, based on Li [15-17] or Nb [21,23] vacancies for the starting pure congruent LN agree that in low doping concentration the divalent ions introduced in the crystal replace niobium on lithium site, Nb$_{Li}$ so-called niobium antisites. In a recent modelling of the various phases of the substitution process in Mg doped LN crystals [23], based on the Nb vacancies model suggested by Abdi et al. for pure undoped LN from congruent to stoichiometric compositions [24], we have shown that in this low concentration range, this substitution process is accompanied by a decrease of Nb vacancies and a change in the Li vacancies to ensure the condition of electrical neutrality of the cell. The first extremum is reached when the total amount of vacancies is minimum. It is to be of note that at this extremum position and above, all cited models describe the substitution and compensation process by the same way. Above this extremum by analogy with phenomena that occurred in Mg doped LN crystals [19], we suggest that Zn ions replace Li ions in their own natural sites up to the threshold concentration corresponding to the lowest limit Li/Nb of the ZnO-LiNbO$_3$ phase diagram. This replacement is accompanied by the creation of lithium vacancies for charge compensation. Finally, when we consider the incorporation of Zn in the high-concentration range above the threshold, the structural data have shown that the most probable compensation mechanism in this case is the self-compensation of [ZnNb]$^{3+}$ by [Zn$_{Li}$]$^{-}$ which
required a concentration ratio Zn_{Nb}/Zn_{Li} = 33\% to conserved the charge neutrality [15]. In this range, the incorporation of Zn ions induces a huge diminution of the Li vacancies as confirmed by various authors [15,22,23]. Therefore, the threshold concentration corresponds to a maximum concentration of Li vacancies and it is known that the photocurrent and the photorefractive properties depend on the concentration on lithium vacancies.

After this description, we can correlate the dependencies of the photorefractive properties on the defect structure of LN:Zn. Nevertheless, we have first to consider the concentration of Zn ions in crystals. The crystal Zn-1 has a dopant concentration, which corresponds to the extremum concentration herein mentioned, whereas the highly doped crystal, Zn-4 is at the threshold concentration. For low light intensities (I < 1MW/m²), as Zn ions only occur as Zn^{2+}, it is allowed that they do not directly contribute to charge transport processes responsible of the photovoltaic effect and of the bulk photoconductivity, which is mainly dominated by Fe^{2+} ions. Nevertheless, at high light intensities (I ≥ 1MW/m²), as used for this study, the previous one center model [15] is not yet available and additional contributions to the photoconductivity and to the bulk photovoltaic effect due to Zn ions and to charge compensating vacancies occur. Thus, the photoconductivity is due to competition effects originate from niobium antisites and lithium vacancies, which means a major influence of substitution phenomena that occurs in the lithium site. At low Zn concentrations, a decrease of the photoconductivity is linked to the decrease of niobium in antisite and we can observe in this concentration range an increase of the photorefraction and of the photosensitivity. For medium concentration range (2-3 mol% ≥ [Zn] ≥ 7 mol%), due to the disappearance of niobium antisite and the increase of lithium vacancies, it appears a large increase of the photoconductivity accompanied by a decrease of the photosensitivity. It was shown [25] that at low impurity concentrations, i.e. in the two described ranges above, the photoconductivity is of n-type whereas above the threshold, it becomes dominantly p-type and increases with the increase of the contribution from the hole component with Zn at the expense of a decrease of the concentration of hole traps i.e. V_{Li} [17].

Like observed in undoped LiNbO_{3} crystals, the E/O properties is a competition between the deformation and the polarizability of NbO_{6} octahedra and the conductivity due to Li vacancies. Therefore, we can analyze the role of Zn, as of Mg in Mg-doped LiNbO_{3} on the EO properties, by the consideration of Nb, Li and vacancies amounts in doped crystals, which is similar to a variation of the composition from the congruent up to the stoichiometric ones for pure LN crystals. At low concentration, the E/O coefficient is linked to diminution of the total amount of vacancies and to the decrease of Nb_{Li}. Above this extremum, the creation of new Li vacancies induces an increase of the electro-optic coefficients up to the threshold. In the high-concentration range, the sharply decrease of the lithium vacancies is accompanied by a new decrease of the electro-optic coefficient.

5. Summary
The photorefractive damage of zinc-doped congruent lithium niobate crystals agrees with the existence of a first extremum concentration for Zn doped crystals around 2-3 mol.% and a threshold about 7 mol%. We have shown that doping above the first extremum concentration with divalent Zn ions leads to a significant decrease of the photorefraction with the increase of Zn concentration.

In Zn doped LN crystals with concentrations above 7 mol%, we observed a large photorefractive damage resistant and thus, due to lower growing difficulties, these crystals present a very interesting alternative in some optoelectronic applications compared to the stoichiometric lithium niobate one. Moreover, the electro-optic coefficients in this concentration range are high and thus, these crystals are very suitable for Q-switching applications.

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