FORMATION OF NANO DOMAINS DURING THE FABRICATION OF SPE WAVEGUIDES ON A PPLN CRYSTAL AND ITS IMPACT ON THE SHG EFFICIENCY.

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Résumé

In this paper we present the study of nano domains formation during the fabrication of channel waveguides produced by Soft Proton Exchange (SPE) in periodically poled congruent lithium niobate (PPLN) crystal. The study was performed using complementary experimental methods such as Piezo Force Microscopy (PFM) and Confocal Raman Microscopy (CRM). It shows that the waveguide fabrication process induces the formation of needle like nano domains on the Z⁻ oriented surface that may be responsible for the poor nonlinear behavior of the waveguides.

MOTS-CLEFS: Niobate de lithium, Echange protonique, Polarisation périodique, nano domaines

In this paper we present the study of nano domains formation during the fabrication of channel waveguides produced by Soft Proton Exchange (SPE) in periodically poled congruent lithium niobate crystal (PPLN) [1]. The study was performed using complementary experimental methods such as Piezoresponse Force Microscopy (PFM) and Confocal Raman Microscopy (CRM).

The sample under investigation is a 0.5-mm-thick Z-cut congruent LN wafer. It was periodically poled using the E-field technique with liquid electrodes and a photoresist mask. Proton exchange procedure was carried out in benzoic acid bath with 2.9% lithium benzoate at 300°C for 3 days, using a SiO₂ mask to define channel waveguides along the X axes. We obtained waveguides with width ranging from 4 to 8 μ m on one side of the sample (initial Z⁻ face) and a planar waveguide with a graded index profile, which was determined using the m-lines technique, on the back side of the sample.

Piezoresponse Force Microscopy (PFM) was used to obtain high-resolution images of the static domain structure at the surface of the crystal [2] (Fig. 1).



Figure 1. PFM characterization of the domain structure of a PPLN crystal crossed by a SPE waveguide

From Fig 1a, it can be seen, that the waveguide fabrication process does not modify the structure of the poled area (Z^+ orientation), while, in the originally oriented crystal (Z^- orientation), the waveguide is clearly visible. If one enlarges the picture, little dots appear at the edge of the waveguide (Fig. 1b). They are characteristic of the presence of surface nano domains [3].



Fig. 2 : Nano domains at the border and in a SPE waveguide realized on the Z^{-} surface.

Going to the ultimate resolution, it is possible to visualize them as well as the dense structure of isolated nano domains in the waveguide with typical lateral sizes of about 50 nm (Fig.2). It is known that isolated nano domains in CLN possess needle-like shape with a form factor ranging between 50 and 100 [4] (see Fig. 3). Therefore it is reasonable to think that they extend down in the crystal from 2.5 to 5 μ m. Therefore, in the Z⁻ area, all the waveguide is affected by these nano domains. They are not present in the Z⁺ area (Fig 3).



Fig. 3 PFM view of the surface at the interface between a Z+ and a Z- area and a schematic of the cut of this interface showing the shape of the nano domains appearing at the surface of the Z-area.

Raman confocal microscopy (RCM) allows analyzing the structure in the depth [5] and Fig. 4 is a cut of two domains walls along the waveguide. From this picture it is clear that the domain walls reach the surface of the crystal, indicating that the periodicity which is necessary to fulfill Quasi Phase Matching (QPM) conditions is preserved, but more investigation are still to be done to understand the response near the surface in the waveguide area. Indeed, from previous studies [6], we do not expect a modification of the Raman spectrum due to the presence of a SPE waveguide.

Despite the fact that those waveguides presented very low losses (between 0.15 and 0.5 dB/cm) which are record values for this kind of devices, the nonlinear characterization of these waveguide were quite disappointing as the maximum obtained conversion efficiency was as low as $10\%/W.cm^2$, and for most of the waveguides, the SHG spectra (Fig. 5) were quite different from the

theoretical sinc² shape. Further investigations are in process to see whether this degradation of the SHG spectra and the reduction of the conversion efficiency can be correlated with the creation of nano domains in the Z^2 area of the PPLN crystal.



Fig. 5 : Typical degraded SHG spectrum obtained with the waveguides presenting nano domains.

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Références

- [1] M.P. De Micheli, Ferroelectrics, 340, 49-62 (2006)
- [2] M.A. Dolbilov, V. Ya. Shur, E.I. Shishkin, M.F. Sarmanova, E.V. Nikolaeva, S. Tascu, P. Baldi, M.P. De Micheli, *Ferroelectrics* 374, 14-19 (2008)
- [3] M.A. Dolbilov, E.I. Shishkin, V.Ya. Shur, S. Tascu, P. Baldi, M.P. De Micheli, *Ferroelectrics*, **398**, 108-114 (2010)
- [4] V.Ya. Shur, Journal of Materials Science, 41(1), 199-210 (2006)
- [5] V. Ya. Shur, P. S. Zelenovskiy, M. S. Nebogatikov, D. O. Alikin, M. F. Sarmanova, A. V. Ievlev, E. A. Mingaliev and D. K. Kuznetsov, J. Appl. Phys. 110, 052013 (2011)
- [6] A. Harhira, Y. Zhang, P. Bourson, L. Guilbert, M. D. Fontana, M. P. De Micheli , Ferroelectrics 01/2007; 352(1):153-157