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RESEARCH ARTICLE

SYNTHESIS AND CHARACTERIZATION OF YTTRIUM DOPED LITHIUM NIOBATE POWDERS

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ABSTRACT

In this research, two types of $LiNbO_3$ congruent composition, doped and substituted with different concentrations of Yttrium (Y) ranging from 0 to 5 mol % are prepared by the classical solid method. The structural properties and composition of the obtained powders are characterized by X-ray diffraction (XRD) and by the Energy Dispersive X-Ray Spectroscopy (EDS). The main results of this work point out the fact that the ceramic powder processing method is a well adapt method for obtaining good quality LN: Y ceramics in the Yttrium concentration range analyzed for both kinds of LN:Y, as the $LiNbO_3$ phase is lonely present in the ceramics at the end of the synthesized process. Lattice Parameter ***c*** increases with Y concentration whereas ***a*** parameter shows constancy for all powders. EDS results show that for substituted powders, Y content in the lattice cell increases monotonously, whereas for the doped ones, Y content increases up to 2.5% and remain constant above this concentration. A mechanism of Yttrium incorporation into the lattice of LN is proposed for the range of doping concentrations for both kinds of studied LN: Y.

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INTRODUCTION

Lithium niobate ($LiNbO_3$, LN)crystal is widely studied and used in optical, laser and communications industry, thanks to its high electrooptic, piezoelectric, acousto-optic and nonlinear optical coefficients, transparency in the wavelength range from near UV to far infrared, large photovoltaic effect and photocurrents in doped crystals. However, when LN crystals are grown from the melt, even with stoichiometric compositions it presents a large Li_2CO_3 deficiency up to 4% (Abdi *et al.*, 2006). Hence LN exists in a wide composition range from congruent the stoichiometric. Therefore, a large amount of defects due to anti-sites defects – Nb in Li sites and structural vacancies necessary to obtain the charge neutrality – is present in non-stoichiometric crystals (Jermann *et al.*, 1994). As a consequence, the LN lattice is a host for incorporation of dopants such as rare earth (RE) or metal ions, even in large concentration (Volk *et al.*, 1994; Volk *et al.*, 1996). Indeed, it is known that the physical properties of LN are strongly dependent of both the intrinsic defects related to the non-stoichiometry and extrinsic defects due to impurities or dopants. Accordingly, several investigations have focused on the LN synthesis and physico-chemical characterization as function of the composition in pure LN and as function the nature and the concentration of single or co-doped LN in order to improve its performance in high-tech applications, including the photorefractive, non-linear optic and laser ones (Novoselov *et al.*, 2010; Abarkan *et al.*, 2008; Zhong *et al.*, 1980; Kokanyan *et al.*, 2004; Minzioni *et al.*, 2007). It's also known that doping or co doping with Holmium (Ho), Erbium (Er) or Ytterbium (Y), allows an efficient up-conversion that could find many new applications (Yi *et al.*, 2004; Lira *et al.*, 2007; Lahoz *et al.*, 2011; Lisiecki *et al.*, 2004; Deren *et al.*, 2006). Hence, this perovskite is a good matrix for the transition elements or the (RareEarth) RE ions, which when doped into the structure, replaces Li^+or/and Nb^{5+} depending on the dopant concentration, type and the synthesis procedure. The doping of lithium niobate material is mainly adding the dopants in the form of oxides to the starting precursors. Many doping were performed with Mg, Fe, Mn, Cr ...., but also with rare earth

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such as Ho, Nd, Tm to optimize particular functional physical properties of LN. These studies show that the incorporation of these elements in the lattice for small percentages (Fadil *et al.*, 2011; Klauer *et al.*, 1992). However, partially all published studies deal with crystals or powders with extrinsic impurities obtained by doping. In principle these ions may occupy any of the three available sites of the octahedron (Li, Nb or a vacant octahedron) or even an interstitial site. As the radii of Nb^{5+} is close to the Li^{+} one from a qualitative reasoning and low concentrations, we can expect that the divalent ions (Mg^{2+}, Fe^{2+}, Cu^{2+}) fall into the Li site structure and that the trivalent ions (Ho^{3+}, Fe^{3+}, Cr^{3+}) incorporate, in an equiprobable manner both Li and Nb sites (with a possible charge of self-compensation) (Nassau, 1967). Beyond a critical concentration, we cannot qualitatively predict the modes of incorporation. LN, as generally used in optical or acoustic devicesare grown from a congruent melt with (Li)/(Nb) = 0.942. For this ratio, the compositionin the crystal is the same as in the melt. The Li deficiency in congruent and off-congruent crystals is accompanied by a creation of intrinsic defects, which depends on their composition. Thus, the relative excess of Nb introduces Nb anti-site defects on Li-sites leads to Li- or Nb-vacancies owing to the charge compensation. These intrinsic defects can be compensated, up to a particular concentration, by adding extrinsic defects. The objective of this work is the study of the impact of the Yttrium on the structural properties of Lithium Niobate and the mechanism of its incorporation into LN cell. The Yttrium is a transition metal, with a similar chemical behavior as the rare earth metals, and usually exists as a trivalent ion, Y^{3+}. We chose to study two series of congruent LN composition called substituted and doped with Yttrium (LN: Y) at different concentrations (up to 5% mol). The phase content and lattice parameters of powders were characterized by X-Ray Diffraction (XRD) and X-ray energy dispersive spectroscopy (EDS).

Experimental Procedure

Y^{3+} doped lithium niobate (LN:Y) powders were prepared by the classical solid state route from high-purity (99.99% from AgfaAesar or Aldrich) niobium oxide Nb_2O_5, lithium carbonate Li_2CO_3 and yttrium oxide. A series of (doped) powders from the congruent melts (with a fixed ratio (Li)/(Nb) = 0.944) and various Y_2O_3 concentrations (0.25, 0.5, 0.75, 1, 1.25, 1.5, 1.75, 2, 2.25, 2.5, 2.75, 3, 4 and 5mol%) were synthetized by the solid-state method. Another series of (substituted) powders from melts with different (Li)/(Nb) ratio ((Li)/(Nb) $\leq$0.944), and Y_2O_3 concentrations determined, to achieve the same Y concentrations above, were grown by the same method. Starting materials were mixed and milled over 30min before a heating treatment according to flowchart and diagram presented in Figure 1 and Figure 2 respectively.

RESULTS AND DISCUSSION

Structure characterization of LN: Y

The crystal structure of the as-synthetized powders was investigated by XRD analysis. The powder X-ray pattern was recorded for all samples (doped and substituted) with various Yttrium concentrations by using a Brucker D8 X-ray diffractometer equipped with a graphite monochromatized CuKα radiation. We recorded the XRD spectra in the 2θ range of 20 to 120° with an increment of 0.01° and a scan speed equal to 0.1 sec/step.

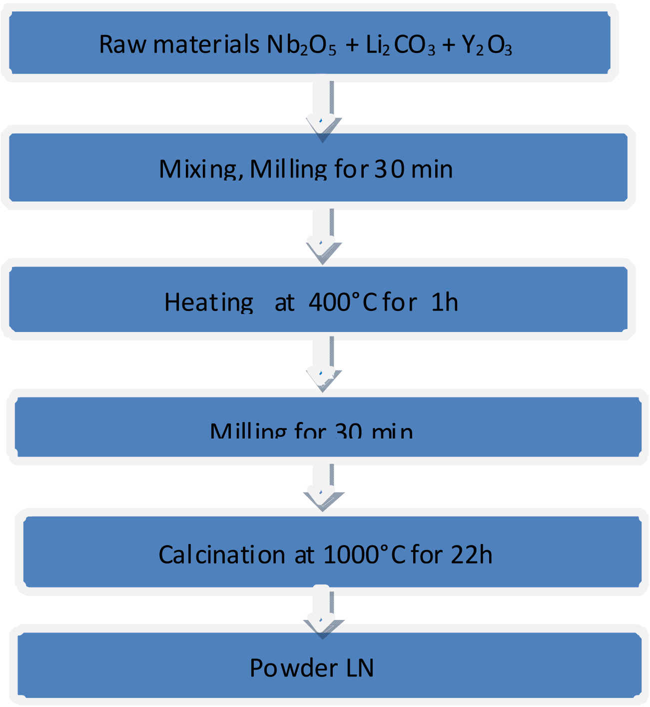

Figure 1. Flowchart of the solid state method used for the preparation of LN:Y powders

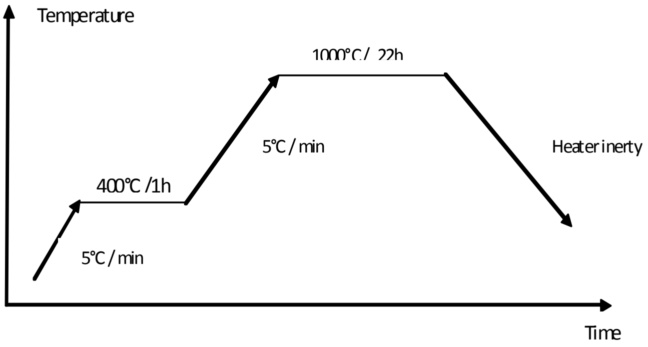

Figure 2. Thermal Cycle used for the calcinations of LN: Y powders

The window aperture is equal to 20mm and the sample holder has a rotation speed of 15 rot/min. Figures 3 show the XRD patterns for both kinds of samples, doped and substituted respectively. These diffractograms show well-resolved peaks, which are a clear signature of the good crystallinity of the particles. These peaks are indexed according to the JCPDS cardsand assigned in pure sample, i.e. (Y) = 0 to the perovskite structure with the hexagonal phase.It is also the case in all doped samples in which Yttrium in the LN structure appears in the patterns with four additional peaks (at 2θ= 28.75°, 30.29°, 47.95° and 49.22°) assigned to a secondary phase $YNbO_4$, for the doped and the substituted LN:Y composition larger than 2mol% of Yttrium. This indicates that Yttrium is completely incorporated into the structure of LN up to 2 mol %.

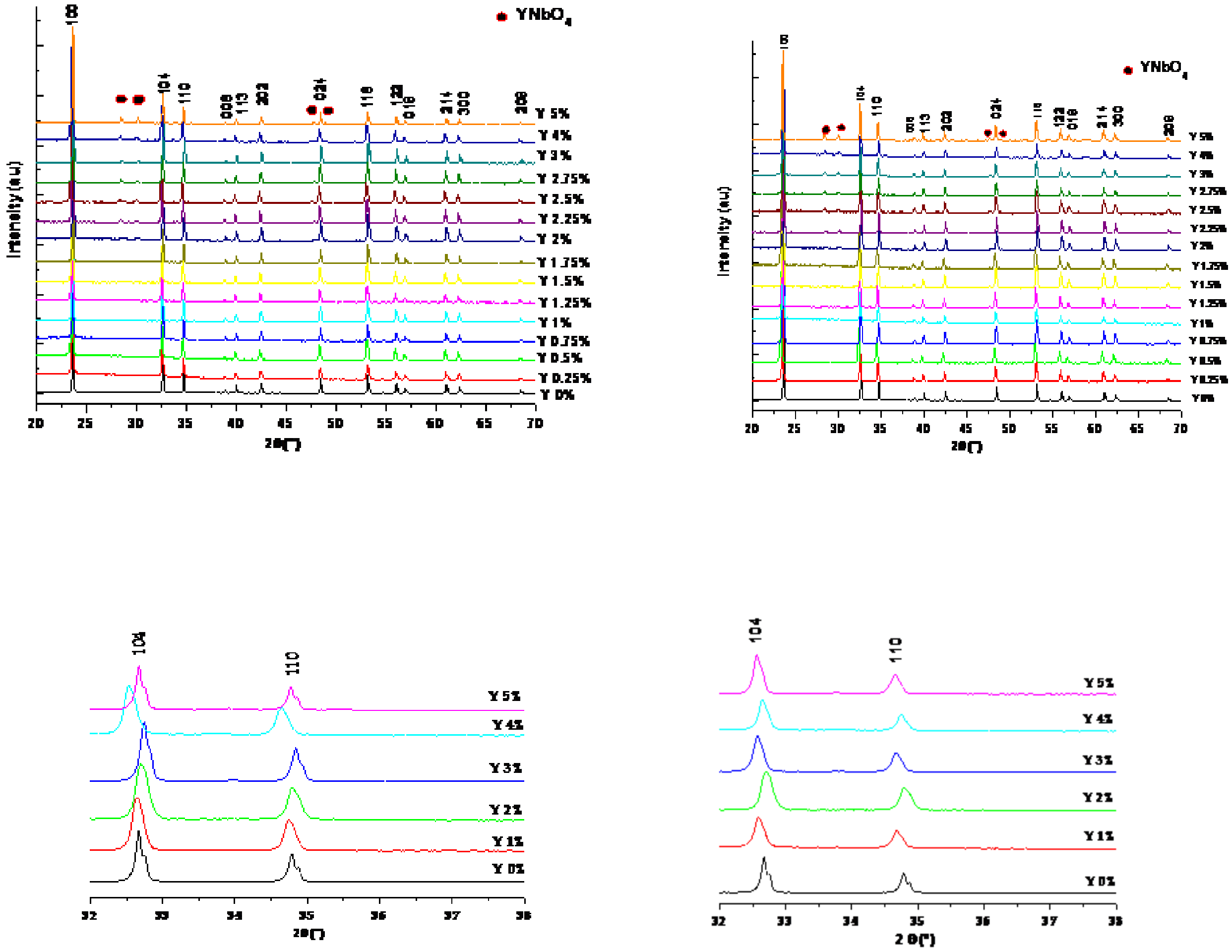

Figure 3. RX patterns of LN:Y doped (Left) and substituted (Right)

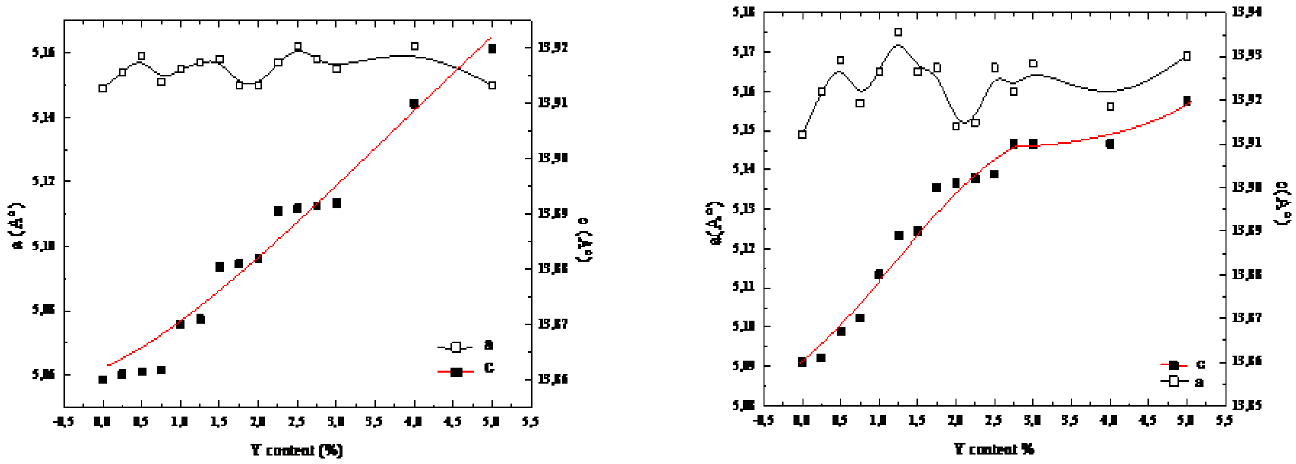

Figure 4. Dependence of lattice parameters a and c on Y contents: of LN:Y doped (Left) and substituted (Right)

Table 1. Energy lines K and L for Nb and Y atoms

Atoms	Energy lines Kα (KeV)	Energy lines Kβ (KeV)	Energy lines Lα (KeV)
Nb	16.568	18.622	2.042
Y	16.733	14.920	1.922

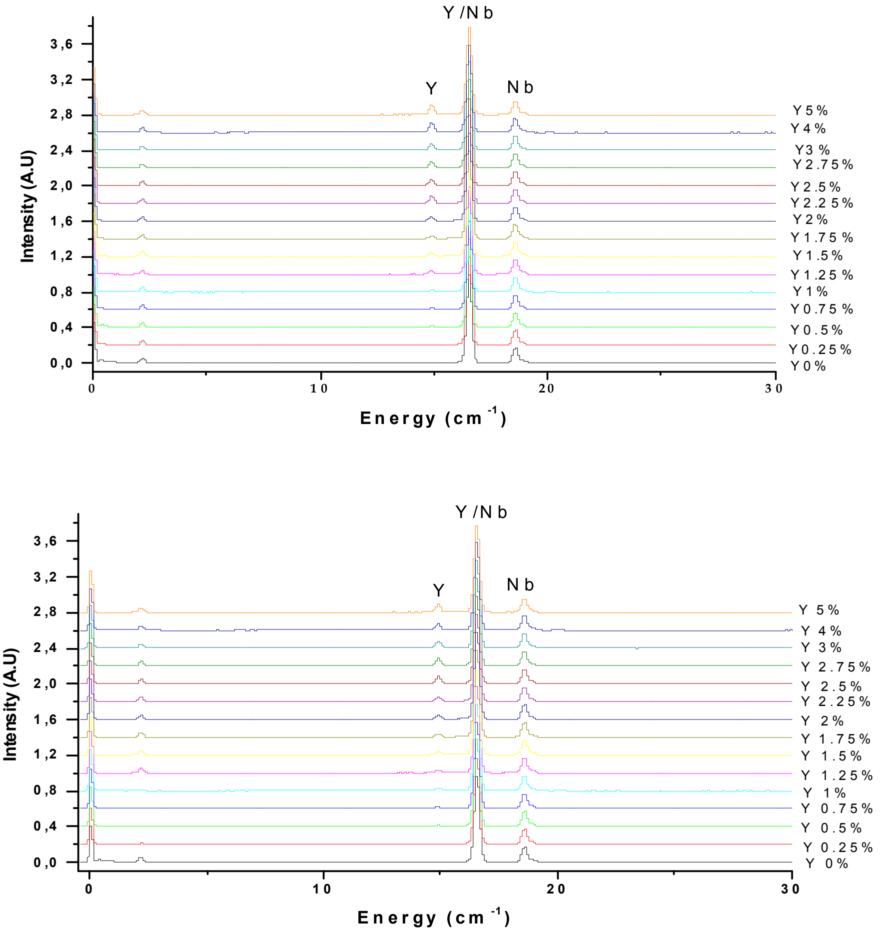

Figure 5. EDS spectra of LN:Y doped (Left) and substituted (Right)

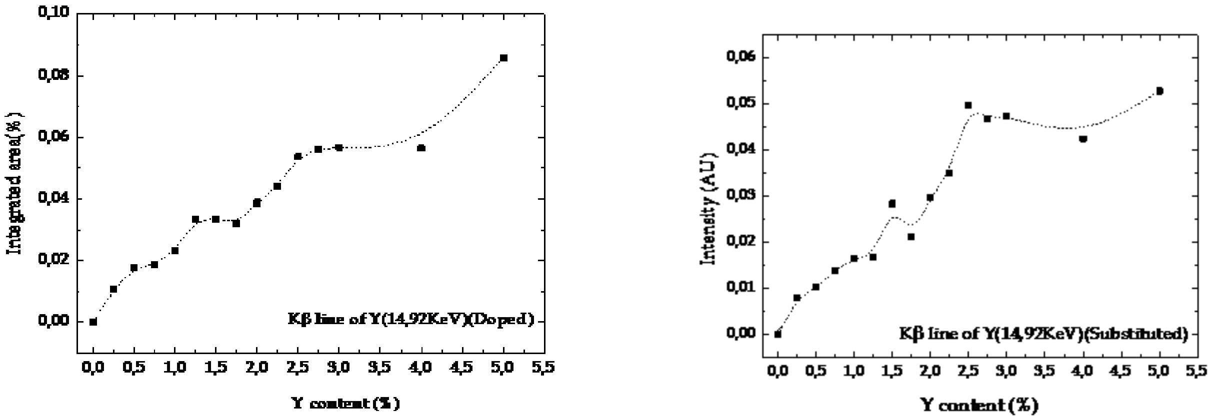

Figure 6. Integrated area, vs. Y concentration, of the EDS K_β line (14.92KeV) of Y peak characteristic of Yttrium in LN:Y : doped (Left) and substituted (Right)

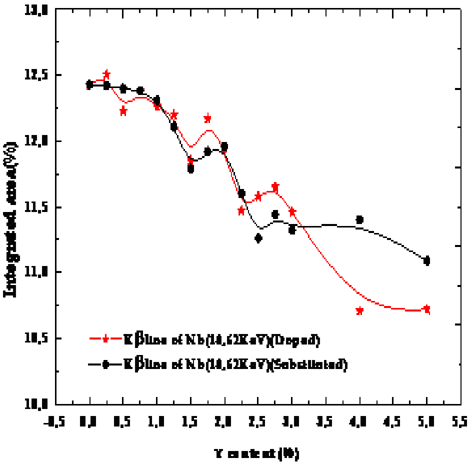

Figure 7. Integrated area, vs. Y concentration, of the EDS K_β line (18.62KeV) of Nb peak characteristic of Niobium in doped and substituted LN:Y

When yttrium content increases we observe a change of the shape and slight shift in the position of some peaks of the XRD spectra (104, 110…) as shown in Figure 3. This result shows that the incorporation of yttrium affects the lithium niobate structure. To study the effect of yttrium on the structure of LN we determined cell parameters **a** and **c** of hexagonal lattice from data of XRD peaks intensities. We have reported the evolution of these lattice parameters with Yttrium concentration in all samples in Figures 4 for doped and substituted powders.

The plots clearly show that, for both types of powders, the lattice parameter a remains slightly constant (around 5.15(5.16) Å) when the concentration of yttrium increases, which is in good agreement with the values reported in the literature (Yu and Xingquan Liu, 2007; Palatnikova *et al.*, 2006). On the other hand, the lattice parameter **c** increases linearly with increasing Y content in LN. These results are in good agreement with the Nassau and Lines model (Nassau and Lines, 1970), which assumes an essential increase in the lattice c with negligible increase in parameter a, in Rare Earth elements (REE) doped $LiNbO_3$ and $LiTaO_3$crystals. In the meantime, we have to note that for the doped powders, the increasing of the c parameter is monotonous (up to 5mol % of Y), when for the substituted powders, after 2.5mol % of Y, the parameter c shows a slight increase. The last value (for 5mol% of Y) for both types of samples is c = 13.92 Å. However for 2.5mol% of Y, values of c parameter are 13.88 Å and 13.9 Å respectively for Y doped and substituted powders.

The difference in values and behaviors of c parameter with Y concentration in LN, and the particular composition LN:Yof 2.5 mol % of Y could be due to the fact that Y_2O_3doping in $LiNbO_3$ has a definite solubility limit. The 2.5mol% Y_2O_3 concentration is close to this solubility limit, for the substituted powders, which contain less Li ions than doped ones, so that the Y dopant is hard to get into the $LiNbO_3$ ceramic. To support this hypothesis, we analyzed our samples by Energy Dispersive X-Ray spectroscopy (EDS).

Energy Dispersive X-ray Spectroscopy

This technique is used for element analysis of our samples LN:Y. Both series of compounds studied LN (doped and substituted) were analyzed. The chemical analyses evaluated by EDS of all the powders are shown in Figure 5 for both doped and substituted samples. All the characteristic lines of heavy chemical elements Nb and Y are observed. The observed peaks correspond to Kα, Kβ and Lα lines of Nb and Y atoms (Table III.1). The direct determination of Lithium by EDS is practically impossible due to the extremely low fluorescence yield of such a light element. The EDS spectrum showed the presence of niobium and Yttrium peaks confirming the incorporation of the Yttrium doping element in all samples. The patterns show that the intensity of the peak characteristic of the Yttrium atom increases with its concentration which is in agreement with the experimental study. I an attempt to understand the relative evolution of Yttrium and Niobium contents in the samples, we determined the integrated areas, in normalized spectra, of resolved and separated peaks characteristic of these two atoms. Results are reported in Figures 6 and 7.

We see that with the concentration of Y, the integrated area of the K_β line (14.92KeV) of Y increases continuously up to 5% of Y in the doped samples. For the substituted samples, the integrated area increases up to a saturation concentration value around 2.5 mol. % of Y and remains slightly constant after this threshold value.

Figure 7 shows the dependence of the integrated area of the K_β line (18.62KeV) of Nb peak on the Y concentration in the samples. In addition, it indicates that for the doped samples, the content of Nb decreases continuously, when for the substituted samples, it decreases below the threshold concentration of Y 2.5mol% and remains slightly constant above this concentration.

DISCUSSION

The results reported in this paper clearly point out a two-type behavior in the Y^{3+} concentration dependence of the structural and fluorescence properties of the LN:Y powders. In the doped LN:Y samples, the results indicate that the concentration of Yttrium increases continuously into the powders. According to figures 4,6 and 7, increasing the Y_2O_3 doping content causes the effects of the continuous increasing of the ***c*** lattice parameter and the diminution of the Nb content in the powders. When for the substituted samples, these parameters reach a value and remain constant, even with increasing the Y_2O_3 content. This occurs at 2.5 mol% doping rate. On the basis of these observations we try to derive a doping mechanism of Yttrium into LN. In LN:Y (doped or substituted), the lattice ***c*** constant increases with the Y concentration because the Y-ion radius (177.6pm) is larger than that of Li-ion(152pm) and Nb-ion(92.908pm) radius. Thus, when Y goes into $LiNbO_3$, Y^{3+} ions are substituted for Li^+ and/or Nb^{4+} ions. Regardless of whether the Y is substituted for Li or Nb cations in LN, the ***c*** lattice parameter of LN:Y would increase (Figures 4). Incorporation of Y in LN is described with the help of appropriate vacancy models. The most commonly used models are the lithium vacancies (Li-site vacancy) and niobium vacancies (Nb-site vacancy) models (Kuz'minov, 1987). A number of works are in favor of each of the two models. In a previous work (Abdi et al., 2006), we reported that the congruent composition is well described by the Nb-vacancy model, and proposed a defect model for the Mg doping of LN (Abdi *et al*., 2009). We showed that the insertion of the Mg ions into the Li position was accompanied by a decrease in the concentration of intrinsic Nb_{Li} defects up to a threshold, and below this concentration, the Mg atoms change their locations in the lattice and partially occupy the Nb positions. In the following we consider the Nb-site vacancy model. Based on this model and our experimental results, an intrinsic defect evolution model is proposed.

The congruent composition is described by the formula:

$$Li_{1-5x}Nb_{5x}]_{Li}Nb_{1-4x}VNb_{4x}]_{Nb}O_3 \quad \dots\dots\dots\dots\dots\dots..(1)$$

Where Nb* are antisite Nb_{Li} and VNb are the Nb vacancies, with x=0.00935 chracterize the Li deficiency (5x) corresponding to a ratio for a composition (*Xc*, in the range 48.3%–48.6% are considered in literature for the pure congruent composition of LN crystals (Donnerberg *et al*., 1996; Iyi *et al*., 1995; Grabmaier and Otto, 1986; Bordui *et al*., 1991). Now we consider incorporation of Y ions into the pure samples described by the chemical formula above. It seems rather well established from literature that rare-earth (RE) divalent and also trivalent ions are predicted to occupy the Li^+ (A)sites by replacing Nb_{Li} antisites (Abdi *et al*., 2009; Iyi *et al*., 1992; Liu *et al*., 1996; Donnerberg, 1996; Lorenzo *et al*., 1995) until all Nb_{Li} ions were replaced. After that RE ions change their locations in the lattice and partially occupy the Nb positions. This occurs at a threshold concentration of the RE ion, which manifests itself as singularities of physical properties in the concentration dependences. So in our case, when Y is doped into the congruent LN crystals, the Y ions replaced first the antisite Nb_{Li} ions whom are pushed toward their natural sites until all Nb_{Li} ions are replaced. The incorporation of Y into LN is charge compensated by a decreasing of the Nb vacancies according to the formula:

$$Li_{1-5x}Nb_{5x-\beta}Y_{\beta}\Big]_{Li}Nb_{1-4x+\frac{2}{5}\beta}VNb_{4x-\frac{2}{5}\beta}\Big]_{Nb}O_3 \quad \dots\dots\dots\dots.(2)$$

Where β is the rate of Y into the powders. At the end of this sage, all Nb_{Li} are replaced by Y, this occurs for β=5x. It corresponds to a concentration (Y) = 2.3mol % and leads to the formula:

$$Li_{1-5x}Y_{5x}]_{Li}Nb_{1-2x}VNb_{2x}]_{Nb}O_3 \quad \dots\dots\dots\dots\dots\dots\dots\dots\dots.(3)$$

We note that this threshold concentration is in agreement with the reported thresholds for trivalent ions found about 2.5 mol% (Kong *et al*., 2004). For concentrations of Y above 2.3mol% of Y, the remaining intrinsic Nb-vacancies are progressively consumed through accommodation of additional Y ions according to the chemical formula;

$$Li_{1-5x}Y_{5x}]_{Li}Nb_{1-2x-\frac{3}{5}\beta}Y_{\beta}VNb_{2x-\frac{2}{5}\beta}\Big]_{Nb}O_3 \quad \dots\dots\dots\dots\dots\dots(4)$$

At the end of this second stage, all Nb-vacancies are fulfilled; this leads to the very simple chemical formula:

$$Li_{1-5x}Y_{5x}]_{Li}Nb_{1-5x}Y_{5x}]_{Nb}O_3 \quad \dots\dots\dots\dots\dots\dots\dots. (5)$$

The Y concentration at this end of stage is (Y) = 4.67%.

For our substituted samples, with increasing Y rate, the number of Li^+ ions in Li sites falls down, and at the Y concentration of 2.5mol% (in the raw materials), this ration is very close to the lowest limit (0.86 in the crystal) of the LN:Mg phase diagram (Grabmaier and Otto, 1986; Hu *et al*., 1991). Thus Y ions couldn't integrate LN powder; and the incorporation process ends at the formula of equation (3). This is because the Y_2O_3 doping concentration has reached to incorporation limit and Y concentration remains constant which appears on ***c*** parameters and EDS spectra (Figures 4, 6 and 7).

Conclusion

In this study, doped and substituted $Y_2O_3^-$ $LiNbO_3$ powders were successfully synthetized by the solid-state method, at different concentrations. XRD patterns confirmed the formation of single phase compounds powders for both type of powders. The ***a*** cell parameter remains constants with increasing Y concentration for all powders when the ***c*** parameter increases with Y content for doped samples. For substituted samples the *c* parameter increases and reaches a maximum value at 2.5 mol% of Y_2O_3. EDS study shows that Yttrium ions incorporate LN powders. The integrated area of the EDS peak characteristic of Yttrium increases continuously for doped samples, but for substituted sample it reaches a maximum value at 2.5 mol% of Y_2O_3 and remains constant after this concentration. The integrated area of the EDS peak characteristic of Niobium decreases continuously for doped samples but reaches a minimum value and remain constant after 2.5 mol% of Y_2O_3 in substituted samples. These experimental results are interpreted on the basis of niobium-vacancy model used to describe the pure congruent composition. In our interpretation, we stipulate that, in a first stage, for the doped powders as long as the molar doping rate is smaller than 2.5 mol. % Y and provided the amount of Nb vacancies necessary for charge compensation allows it, the Y^{3+} ions replace gradually antisite Nb^{5+} cations at Li sites. Charge neutrality is insured by a decreasing of niobium-vacancies. In a second stage, the remaining Nb vacancies are filled by Y ions up to 4.67 mol.% Y. At this point, the number of vacancies null. For the substituted powders the doping process ends at the first stage because the samples are highly Li deficient so that the lowest limit of the ratio is reached and it is to hard to incorporate Y into LN . Hence after 2.5mol% Y, Y, Nb contents, and thus the ***c*** parameter remain constant.

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