

# Microstructural Characterization of LiNbO<sub>3</sub> and LiNbO<sub>3</sub>:Mn Nanofibers Synthesized by Electrospinning

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In this work, we synthesized pure and Mn-doped LiNbO<sub>3</sub> nanofibers by the electrospinning method and fully characterized the nanostructures by SEM, DRX, TEM and Raman techniques and compared experimental Raman results with ab initio calculations, to probe lattice site substitution of Mn ions.

For pure LiNbO<sub>3</sub>, the precursor solution consisted of poly(vinylpyrrolidone) (PVP), niobium ethoxide (Nb(OCH<sub>2</sub>CH<sub>3</sub>)<sub>5</sub>) and lithium hydroxide (LiOH), dissolved in acetic acid CH<sub>3</sub>COOH and ethanol C<sub>2</sub>H<sub>5</sub>OH. The ratio of molar composition of lithium hydroxide niobium ethoxide was set to 1:1. On the other hand, for Mn-doped LiNbO<sub>3</sub> nanofibers, the precursor solution consisted of lithium hydroxide LiOH, niobium ethoxide Nb(OCH<sub>2</sub>CH<sub>3</sub>)<sub>5</sub>, manganese acetate Mn(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub> and poly(vinylpyrrolidone) (PVP), dissolved in acetic acid CH<sub>3</sub>COOH and ethanol C<sub>2</sub>H<sub>5</sub>OH. The ratio of molar composition of lithium hydroxide niobium ethoxide and manganese acetate was set to 1:1:0.025.

In both cases, the solution was heated at 25°C while being stirred during 2 hours and then delivered into a metallic needle at a constant flow rate of 0.3 mL/h by a syringe pump. With an applied high-voltage of 15 kV, producing an electric field of 1x10<sup>5</sup> V/m, the precursor solution jet was accelerated toward the aluminum foil, leading to the formation of LiOH/Nb(OCH<sub>2</sub>CH<sub>3</sub>)<sub>5</sub>/PVP and LiOH/Nb(OCH<sub>2</sub>CH<sub>3</sub>)<sub>5</sub>/Mn(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub>/PVP fiber composites, for pure and Mn-doped respectively, together with a rapid evaporation of the ethanol.

For the calculation of the phonon modes, the LiNbO<sub>3</sub> structure was represented by a 2x2x2 supercell, with 80 atoms, and the structure was fully relaxed to its equilibrium configurations until the Hellmann-Feynman forces were smaller than 0.001 eV/Å. The Hellmann-Feynman forces were then calculated for displaced atoms, one at a time.

By comparing experimental Raman spectra of pure and Mn-doped LiNbO<sub>3</sub>, and the results of the calculations, we were able to probe Mn ion incorporation into the LiNbO<sub>3</sub> lattice and show that Mn substitutes Li sites.

Figs. 1 shows SEM micrographs of as-spun fibers. Cylindrical and randomly oriented Nb(OCH<sub>2</sub>CH<sub>3</sub>)<sub>5</sub>/LiOH/PVP and LiOH/Nb(OCH<sub>2</sub>CH<sub>3</sub>)<sub>5</sub>/Mn(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub>/PVP composite fibers were obtained, with diameters in the range 93-163 nm.

Fig. 2 shows XRD patterns from calcined composite fibers, showing the formation of crystalline samples of pure and Mn-doped lithium niobate. However, the presence of Mn in this structure is confirmed by energy-dispersive X-ray spectrometry (EDS), as shown later.

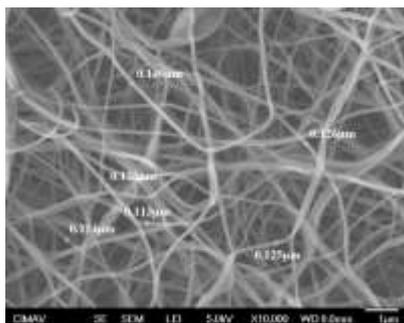
Fig. 3 shows TEM micrographs of necklace-like nanofibers from calcined Mn-doped LiNbO<sub>3</sub>.

Fig. 4a and d shows an EDS mapping through a typical nanofiber, where it can be seen that Mn atoms are uniformly distributed in the structure.

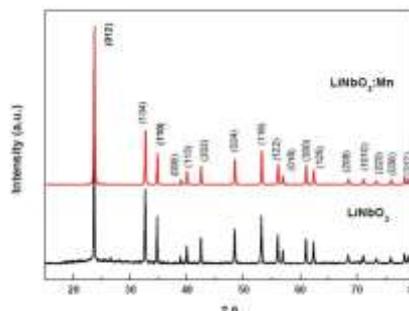
Fig. 5 shows the Raman spectra of the calcined electrospun nanofibers in the spectral range of 100 to 700 cm<sup>-1</sup>, along with calculated LiNbO<sub>3</sub> spectrum.

**References:**

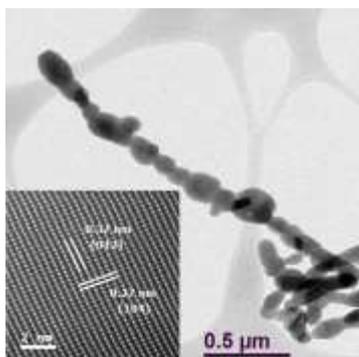
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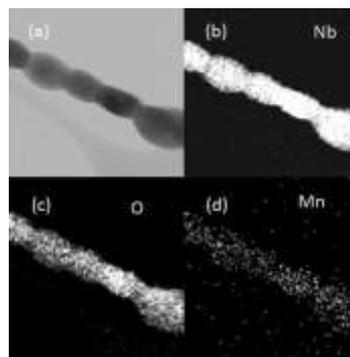
**Fig. 1.** SEM image of as-spun LiOH/Nb(OCH<sub>2</sub>CH<sub>3</sub>)<sub>5</sub>/PVP fiber composite.



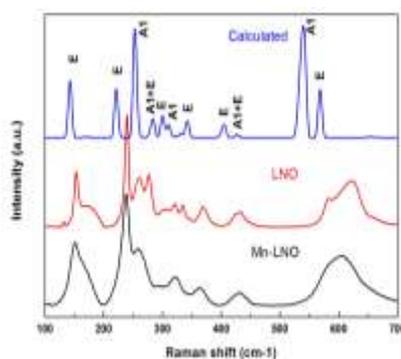
**Fig. 2.** XRD patterns of calcined pure and doped LiNbO<sub>3</sub>.



**Fig. 3.** TEM image of Mn-doped LiNbO<sub>3</sub> nanofibers.



**Fig. 4a-4d.** EDS mapping through a typical nanofiber.



**Fig. 5.** Raman spectra of calculated, pure and Mn-doped LiNbO<sub>3</sub> nanofibers.



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