

Synthesis and Characterization of LiNbO₃ nanofibers

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Ferroelectric oxides ABO₃ in which A is an alkali or rare-earth element, and B represent a transition metals, such as LiNbO₃ with a perovskite structure, are widely used in the fields of nonlinear optics, pyroelectric detectors, electro-optical modulators, thin-film capacitors, and optical memories [1,2]. Because their properties are dependent not only on its chemical composition but also on its structure, shape, and size, it has been found that reduction of the grain size to the nanoscale leads to distinct properties from those of the bulk.

Over the last few decades, one dimensional nano materials such as nanotubes and nanofibers, have attracted great attention due to their unique structure and properties, i.e. large specific surface area and chemical/mechanical stabilities. Thus nanofibers can be used as building blocks in nanotechnology [3,4]. Previously, several ceramic nanowires have been synthesized by various processes, e.g. solution method, laser ablation and chemical vapor deposition (CVD). Recently, there has been an intense research effort on electrospinning of ceramics since it is a straightforward way to synthesize nanostructures.

The synthesis of LiNbO₃ nanofibers was carried out by the electro-spinning method. A detailed description of the procedure can be found in the literature [5]. In this work, the precursor solution was composed of poly(vinylpyrrolidone) (PVP), niobium ethoxide (Nb(OCH₂CH₃)₅) and lithium hydroxide (LiOH), dissolved in ethanol. The solution was heated at 30°C with stirring for 2 hours and then delivered into a metallic needle at a constant flow rate of 0.3 mL/h by a syringe pump. The metallic needle was connected to a high-voltage power supply and a grounded aluminum foil was placed 15 cm from the needle tip. With an applied high-voltage of 13 kV, the precursor solution jet was accelerated toward the aluminum foil, leading to the formation of VOSO₄/Nb(OCH₂CH₃)₅/LiOH/PVP fiber composite, together with a rapid evaporation of the ethanol. The composite nanofibers were then annealed at 700 °C for 2 hour, with a slope of 3°C/min to obtain LiNbO₃ nanofibers.

The presence of pure phase is confirmed by XRD analysis, as shown in Fig. 1, for calcined fibers, showing the formation of crystalline LiNbO₃. Diffraction-peak identification is performed on the basis of the PDF2 release 2010 ICDD database. Fig. 2 shows SEM micrograph of as-spun fibers. Cylindrical and randomly oriented fibers were obtained with diameter about 60-150 nm. Fig. 3 shows TEM micrograph from an isolated, calcined LiNbO₃ nanofiber, after dispersing the sample in isopropanol, with a few μm long, where the nanoparticles composing the fiber are clearly seen. Fig. 4 shows the same fiber at higher magnification, where well defined planes are observed, indicating a good crystallinity.

References

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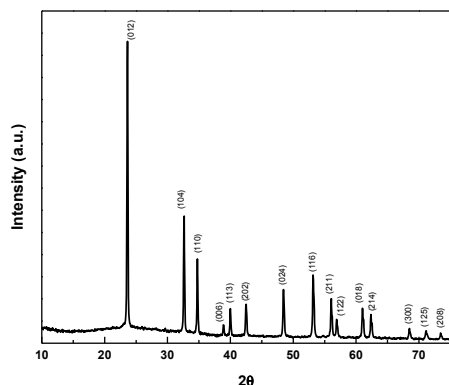


Figure 1. XRD pattern of as-spun VOSO_4/PVP composite.

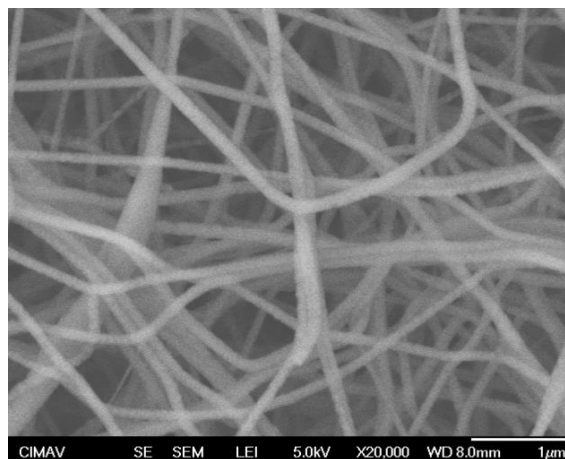


Figure 2. SEM images of as-spun and calcined $\text{VOSO}_4/\text{Nb}(\text{OCH}_2\text{CH}_3)_5/\text{LiOH}/\text{PVP}$ composite.

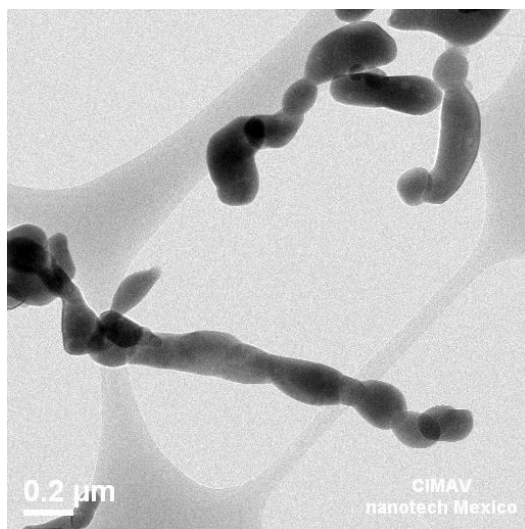


Figure 3. TEM image of LiNbO_3 nanofibers.

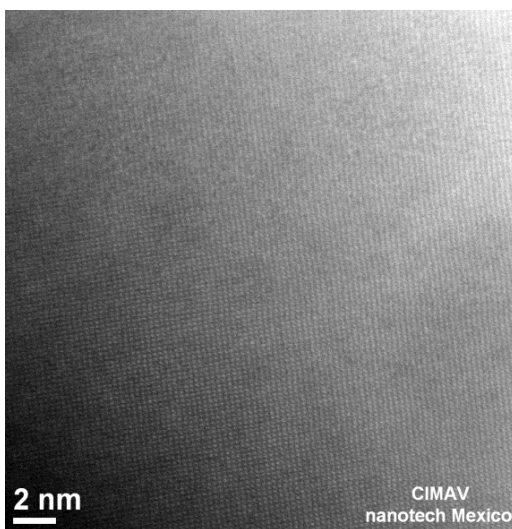


Figure 4. TEM image of a nanoparticle composing the nanofiber.