

Characterization by Raman and Scanning Electron Microscopy of LiNbO₃ Powders Obtained by Mechanochemical and Calcination Route

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Lithium niobate (LiNbO₃) is an electro-optical material with a wide range of applications in modulators, waveguides, frequency doubling, second harmonic generation and high-density storage [1]. The conventional route for producing LiNbO₃ powders is through solid-state reactions at temperatures above 1000°C [2]. Nowadays, mechanochemistry is gaining attention as an alternative to obtain powders of metal oxides with perovskite-type structure [3]. However, the technique has been less effective in obtaining niobates and, consequently, the synthesis must be completed by heating the samples for several hours at high temperatures (800-900°C) [4]. Further, mechanochemistry can be used to increase the reactivity of the precursors, which in turns lowers the temperature for synthesis [4]. Herein we report the synthesis of LiNbO₃ powders obtained by precursor activation using the mechanochemical route followed by calcination at 900°C for 3h. The powders were structurally characterized by Raman and scanning electron microscopy.

Nb₂O₅ (99.5% pure; 100 mesh powder) and Li₂CO₃ (99% pure) were purchased from Alfa Aesar chemicals and used as received. A mixture of 0.7825 g Nb₂O₅ (3 mmol) and 0.2175 g Li₂CO₃ (3 mmol) was ground for 20 min using an agate mortar. A drop of deionized water was added to assist the grinding. The homogenous mixture was placed in a conventional furnace for calcination at 900°C for 3 hours in air. Solid-state Raman spectra were taken in a Horiba Jobin-Yvon LabRam HR high-resolution Raman microscope, equipped with a charge-coupled device detector and an excitation laser source with a wavelength of 632.8 nm. Scanning Electron Microscopy (SEM) analysis was carried out with a FESEM JEOL JSM-7800F, operated at 10 kV, under secondary electron mode.

Raman spectra showed that the ground mixture was mainly composed by Nb₂O₅ phase, which would indicate that the Li₂CO₃ dissolved in the medium due to the water addition and the mechanochemical process (see Figure 1). The latest ensured a homogenous distribution of Li atoms in the medium. By this way, the reactivity of Li atoms was increased and consequently, the diffusion of Li was favoured during calcination at 900°C - 3h giving the formation of LiNbO₃ (see Figure 1). SE images showed an irregular morphology of the LiNbO₃ powders (see Figure 2). The particle surface morphologies are granular, which indicated that the agglomerates are formed by primary particles that fused during calcination. The size of the grains ranges from 0.2 to 1.5 μm.

References:

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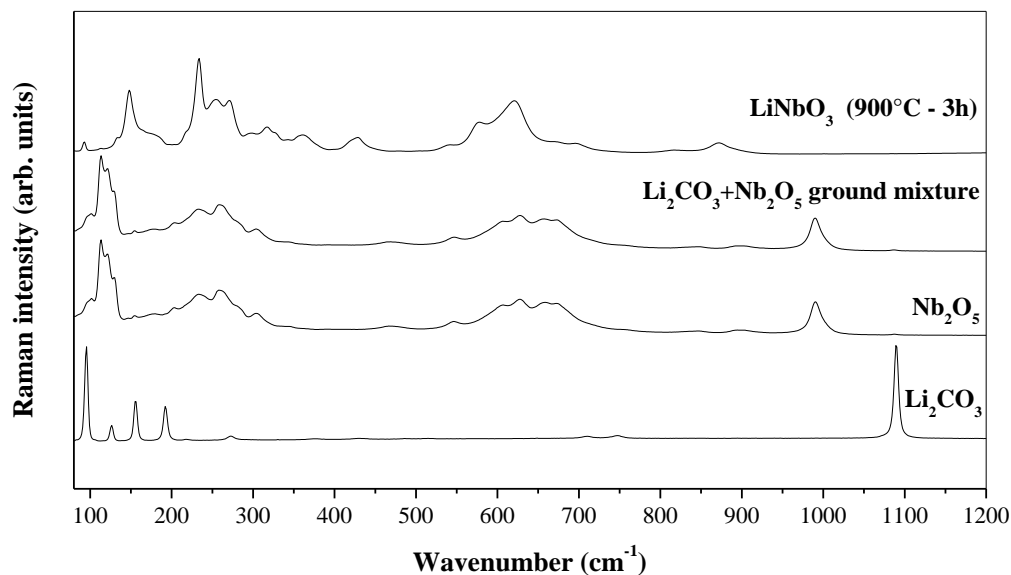


Figure 1. Raman spectra of the precursors Li₂CO₃ and Nb₂O₅ along with the ground mixture Li₂CO₃ + Nb₂O₅ and LiNbO₃ powders after calcination (900°C, 3h).

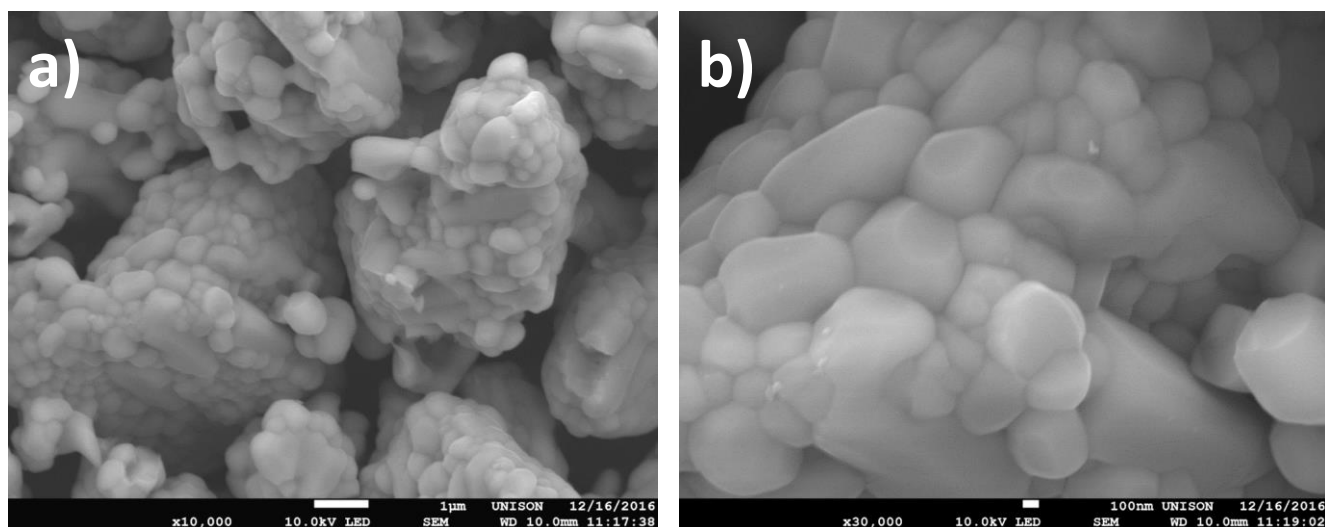


Figure 2. SE images of LiNbO₃ powders after calcination (900°C, 3h).