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Li2o Loss of Lithium Niobate Nanocrystals during High-Energy Ball-Milling

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Abstract : The aim of our research is to prepare rare-earth-doped lithium niobate (LiNbO3) nanocrystals, having only a few dopant ions in the focal point of an exciting laser beam. These samples will be used to achieve individual addressing of the dopant ions by light beams in a confocal microscope setup. One method for the preparation of nanocrystalline materials is to reduce the particle size by mechanical grinding. High-energy ball-milling was used in several works to produce nano lithium niobate. Previously, it was reported that dry high-energy ball-milling of lithium niobate in a shaker mill results in the partial reduction of the material, which leads to a balanced formation of bipolarons and polarons yielding gray color together with oxygen release and Li2O segregation on the open surfaces. In the present work we focus on preparing LiNbO3 nanocrystals by high-energy ball-milling using a Fritsch Pulverisette 7 planetary mill. Every ball-milling process was carried out in zirconia vial with zirconia balls of different sizes (from 3 mm to 0.1 mm), wet grinding with water, and the grinding time being less than an hour. Gradually decreasing the ball size to 0.1 mm, an average particle size of about 10 nm could be obtained determined by dynamic light scattering and verified by scanning electron microscopy. High-energy ball-milling resulted in sample darkening evidenced by optical absorption spectroscopy measurements indicating that the material underwent partial reduction. The unwanted lithium oxide loss decreases the Li/Nb ratio in the crystal, strongly influencing the spectroscopic properties of lithium niobate. Zirconia contamination was found in ground samples proved by energy-dispersive X-ray spectroscopy measurements; however, it cannot be explained based on the hardness properties of the materials involved in the ball-milling process. It can be understood taking into account the presence of lithium hydroxide formed the segregated lithium oxide and water during the ball-milling process, through chemically induced abrasion. The quantity of the segregated Li2O was measured by coulometric titration. During the wet milling process in the planetary mill, it was found that the lithium oxide loss increases linearly in the early phase of the milling process, then a saturation of the Li2O loss can be seen. This change goes along with the disappearance of the relatively large particles until a relatively narrow size distribution is achieved in accord with the dynamic light scattering measurements. With the 3 mm ball size and 1100 rpm rotation rate, the mean particle size achieved is 100 nm, and the total Li2O loss is about 1.2 wt.% of the original LiNbO3. Further investigations have been done to minimize the Li2O segregation during the ball-milling process. Since the Li2O loss was observed to increase with the growing total surface of the particles, the influence of ball-milling parameters on its quantity has also been studied.

Keywords : high-energy ball-milling, lithium niobate, mechanochemical reaction, nanocrystals
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