 ROBL-CRG	Experiment title: Structural modifications of sputtered lithium niobate during in-situ annealing	Experiment number: 20-02 701
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Lithium niobate (LiNbO₃) is one of the most technologically important oxides, with an extraordinary combination of ferroelectric, piezoelectric, acoustic, optical, and also ion conducting properties [1]. In a recent study we investigated how structural disorder modifies ion conductivity and ionic mobility of this material [2]. Annealing and in-situ-crystallization of an initially amorphous sample can be used to produce nano-crystalline samples with different degrees of disorder in a defined way. An absolute prerequisite for reliable studies of ionic mobility as a function of disorder is a systematic characterization of the structural state of samples *identical* to those which will be used for the transport measurements. The aim of the present experiment was to investigate structural modifications of sputtered amorphous lithium niobate during isothermal in-situ-annealing.

Lithium niobate films with a thickness of about 300 nm, which were also used for the investigation of Li tracer diffusion [2], were prepared by ion beam sputtering on sapphire substrates. Measurements at BM20 were carried out by in-situ X-ray Diffractometry (XRD) in the $\theta/2\theta$ mode in the temperature range between 316 and 514 °C in a resistance furnace in a vacuum of about 5×10^{-6} mbar. Fig. 1(a) shows the XRD diagrams recorded at different temperatures. After deposition the material is

amorphous. Crystallization starts at a temperature of 337 °C (annealing time about 1 h). For temperatures up to 415 °C LiNbO_3 , is the only crystallized phase. At temperature above, Bragg peaks corresponding to LiNb_3O_8 are additionally formed, which refers to a decomposition of the stoichiometric phase. An important result is that the integrated area of the Bragg peaks does not change significantly during annealing. From this finding, it can be concluded that nearly complete crystallization of the film occurs already during the first annealing step at 337 °C. At higher temperatures, only the crystallite size increases (Fig. 1(b)) as obtained from an analysis of the peak width using the Scherrer formula. The present grain growth is the consequence of a coarsening process and not of an additional crystallization of an amorphous rest phase. In addition, prolonged isothermal annealing for several hours at a constant temperature does not alter significantly the width of the Bragg peaks, meaning a significant modification of grain size can be achieved only by raising the temperature and not by prolonged annealing. An analysis with the Williamson-Hall method indicates a negligible amount of microstrain to be present in the films during the experiments.

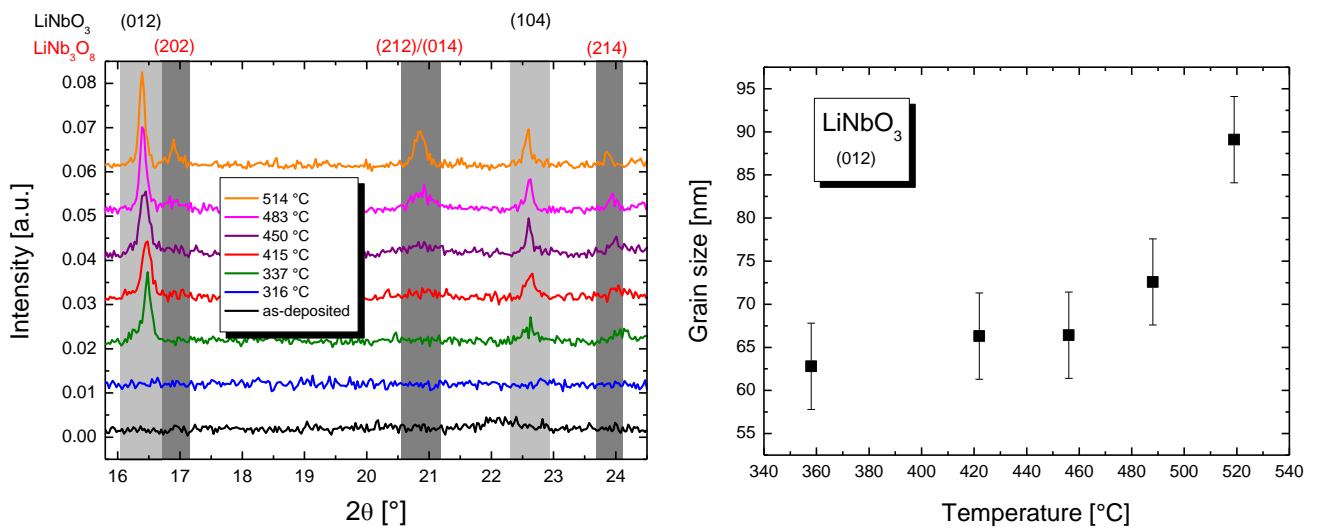


Fig. 1 (a) X-ray diffractograms measured during in-situ annealing of a LiNbO_3 film at various temperatures. (b) Grain size of nano-crystalline LiNbO_3 as a function of annealing temperature (about 1 h of annealing).

[1] R. S. Weis, K. Gaylord, Appl. Phys. A37 (1985), 191.

[2] J. Rahn et al. Z. Phys. Chem. 226 (2012), 439.