



<p><b>Experiment title:</b> The structure and the phase transition sequence of the NaNbO<sub>3</sub> nanocrystals formed in the glass matrix</p>	<p><b>Experiment number:</b> <b>HC-3352</b></p>	
<p><b>Beamline:</b> BM01A</p>	<p><b>Date of experiment:</b> from: 22.11.2017 to: 27.11.2017</p>	<p><b>Date of report:</b> 16.01.2018</p> <p><i>Received at ESRF:</i></p>
<p><b>Shifts:</b> 9</p>	<p><b>Local contact(s):</b> Dr. Chernyshev Dmitriy</p>	

**Names and affiliations of applicants** (\* indicates experimentalists):  
 Vakhrushev Sergey, Laboratory Russian Academy of Sciences Ioffe Physico-Technical Institute  
 Koroleva Ekaterina, Laboratory Russian Academy of Sciences Ioffe Physico-Technical Institute  
 Alekseeva Olga, St. Petersburg state polytechnical university  
 Vanina Polina, St. Petersburg state polytechnical university

**Report:**

The results of X-ray diffraction study of the structure and the phase transition sequence of the NaNbO<sub>3</sub> nanocrystals formed in the glass matrix are reported. The diffraction spectra of ferroelectric glass-ceramics containing niobate-based crystals (glass annealed at temperature 610 °C) were obtained over a wide temperature range (-173 °C – 600 °C) for samples S4 containing 35% of Nb<sub>2</sub>O<sub>5</sub> with different annealing time (206, 21, 8 and 2 hours). Figure 1 shows the diffraction patterns for the sample S4(21h) at two temperatures: 27 and 600 °C. Preliminary analysis showed that at room temperature the structure of all samples corresponds to antiferroelectric phase (*Pbcm* space group) as in bulk samples [1], and also the absence of phase transitions (Figure 1), unlike bulk samples [1] undergoing a number of phase transitions in the investigated temperature range.

In addition, NaNbO<sub>3</sub> nanocrystals in sample S2 (sample containing 31% of Nb<sub>2</sub>O<sub>5</sub>) were grown at three temperatures: 610, 600 and 580 °C. Figure 2 shows changes of intensity of NaNbO<sub>3</sub> Bragg peak during the growth process of NaNbO<sub>3</sub> nanocrystals in sample S2. Analysis showed that the structure corresponds to tetragonal phase (space group *P4/mbm*) as in bulk samples. The average size of NaNbO<sub>3</sub> nanocrystals was estimated. Temperature dependencies of nanoparticle size during the growing process are shown in Fig.3. When saturation is reached the size become temperature independent. Some dependence on temperature observing at the beginning of the growth process can be related with both peculiarities of nanocrystals growth or the errors due to background contribution to the width of small intensity peaks.

Figure 1. Diffraction spectra of s4(21 h) sample at different temperatures

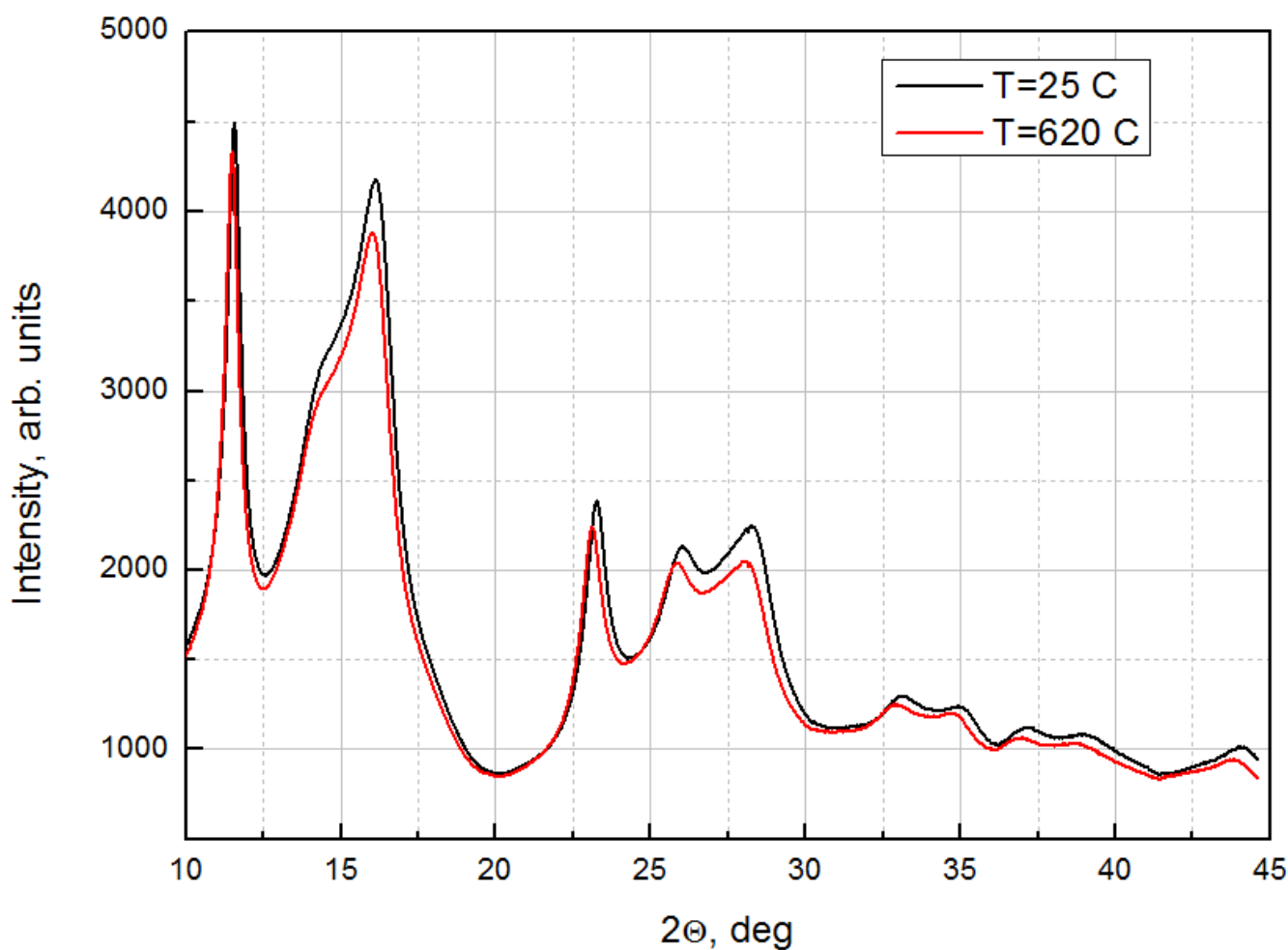


Figure 2. Time evolution of Bragg reflection intensity of NaNbO<sub>3</sub> during growth process at 3 temperatures (s2 sample).

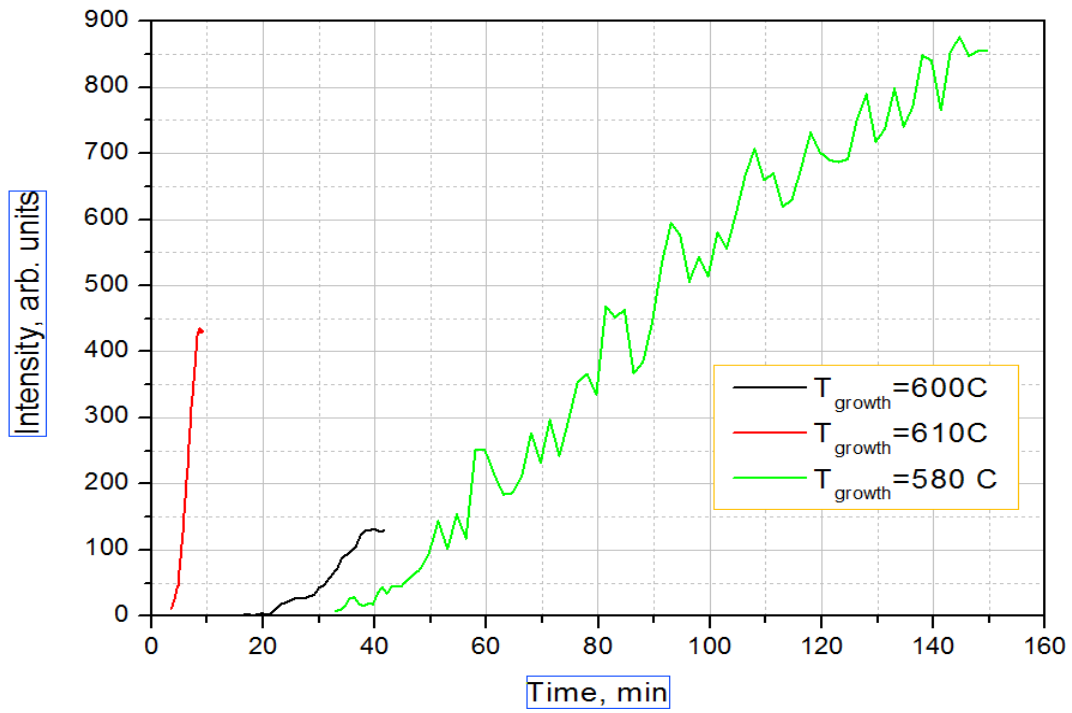
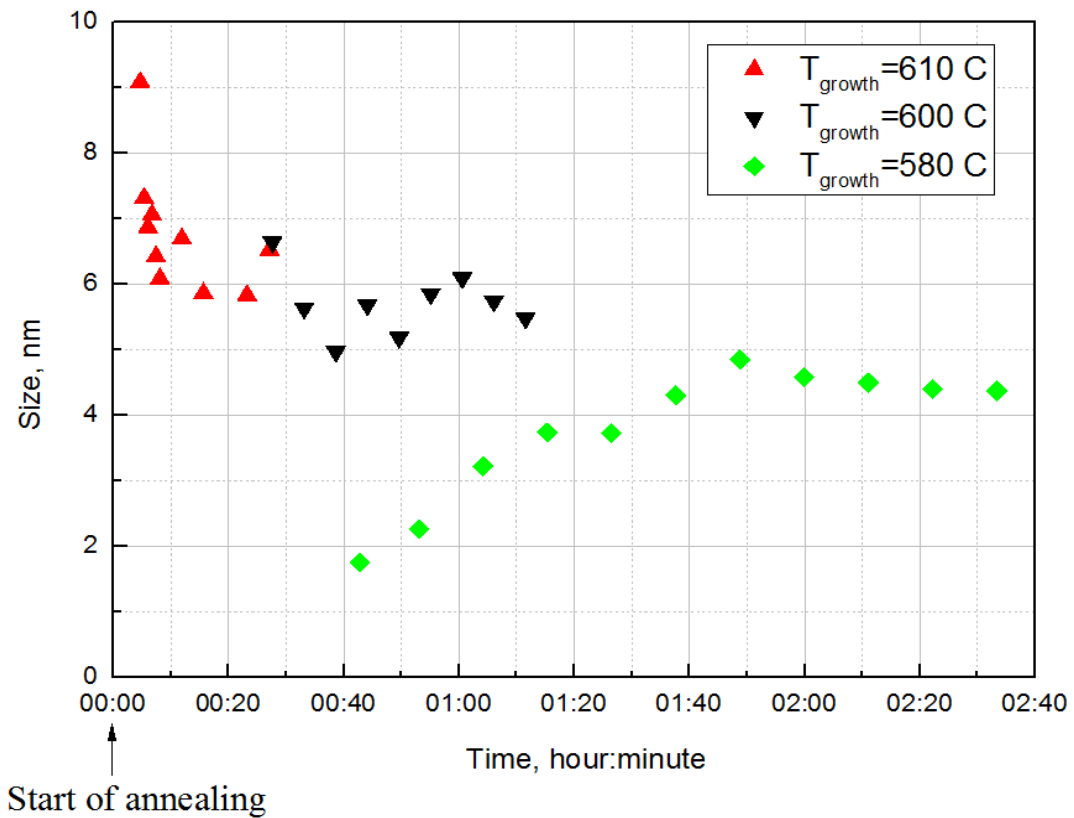


Figure 3. Temperature dependence of  $\text{NaNbO}_3$  nanocrystallites size during growth process (s2 sample).



[1] S. K. Mishra, N. Choudhury, S. L. Chaplot, P. S. R. Krishna, and R. Mittal, Phys. Rev. B 76, 024110 (2007) and references therein.