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Южный федеральный университет
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ФИЗИКА БЕССВИНЦОВЫХ ПЬЕЗОАКТИВНЫХ И РОДСТВЕННЫХ МАТЕРИАЛОВ (АНАЛИЗ СОВРЕМЕННОГО СОСТОЯНИЯ И ПЕРСПЕКТИВЫ РАЗВИТИЯ) (LFPM-2018)

Труды Седьмого Международного междисциплинарного
молодежного симпозиума

г. Ростов-на-Дону – г. Туапсе, 20–24 сентября 2018 года

В двух томах

Том 1

Ростов-на-Дону – Таганрог
Издательство Южного федерального университета
2018

STRUCTURE AND PHASE TRANSITIONS OF ANNEALED $Na_{0.95}Li_{0.05}NbO_3$ POLYCRYSTALLINE SAMPLES

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The results of dielectric, X-ray diffraction and heat capacity studies of the effect of annealing on the properties of $Na_{0.95}Li_{0.05}NbO_3$ (NLN-5) composition have shown a giant increase of T_m exceeding 60 K achieved by annealing at different temperatures within a wide temperature range.

Lead-free $Na_{1-x}Li_xNbO_3$ (NLN) solid solutions are prospective piezoelectronic materials because of a unique combination of parameters, not available in other piezoelectrics. Properties and phase transition temperatures of NLN ceramics depend considerably on the sample's thermodynamic prehistory due to several competing structural instabilities typical of $NaNbO_3$ and its solid solutions [1].

Previously we found out that a long-time (several hours) annealing of NLN ceramics at temperatures below the temperature T_m of the dielectric permittivity ε maximum increases T_m by 20-30 K [2]. However substantial changes of T_m were observed only after annealing in a very narrow temperature range [2, 3].

We carried out dielectric, X-ray diffraction and heat capacity studies of the effect of annealing on the properties of $Na_{0.95}Li_{0.05}NbO_3$ (NLN-5) composition. A giant increase of T_m exceeding 60 K was achieved by annealing at different temperatures within a wide temperature range. However the soaking time necessary to obtain a substantial shift of T_m increased dramatically as the annealing temperature decreased.

The giant shift of T_m determined by dielectric studies of ceramic samples was confirmed by X-ray diffraction and heat capacity studies of NLN-5 powder.

In the as-sintered NLN-5 ceramics dielectric studies give $T_m \approx 360$ °C. This value corroborate the literature data [1] and agrees well with anomalies in the temperature dependencies of the lattice parameters. In the annealed NLN-5 ceramics T_m increases up to ≈ 420 °C.

X-ray diffraction (XRD) experiment was performed using the DRON-7 diffractometer, supplied by the Anton Paar HTK 1200N temperature camera, Co-Kα radiation. Temperature data were collected in the heating mode within 20 - 550 °C temperature range with the step 10 °C.

Fragments of the X-ray diffraction patterns comprising (200), (220) and (222) reflections for the annealed NLN-5 powder are shown on the Figure 1. At temperatures 20 - 390 ° C the reflections are splitted according to the monoclinic distortion of the cubic cell, a=c> b, β>90 deg. (orthorhombic NLN phase).

At further heating the additional component of the (222) reflection appears, testifying the formation of cubic phase; so orthorhombic and cubic phases coexist in the 400-450 °C temperature internal.

At increasing temperature content of orthorhombic phase gradually decreases and above 460°C a pure cubic phase is observed.

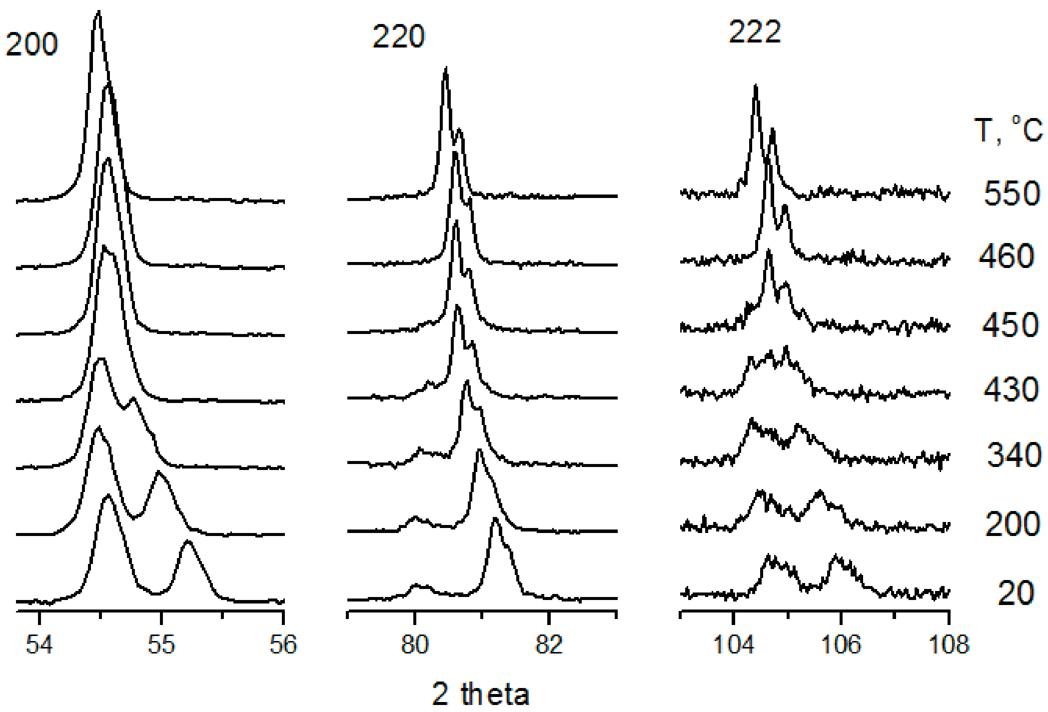

Fig 1. Splitting of (200), (220) and (222) XRD reflections for orthorhombic (20-340 °C) and cubic (460°C, 500°C) phases and phase coexistence region (430°C, 450 °C).

Lattice parameters were calculated with the help of PdWin package. For orthorhombic phase lattice parameters a=c> b, β>90 deg. of subcell with monoclinic distortion were derived from orthorhombic parameters A,B,C (Fig 2).

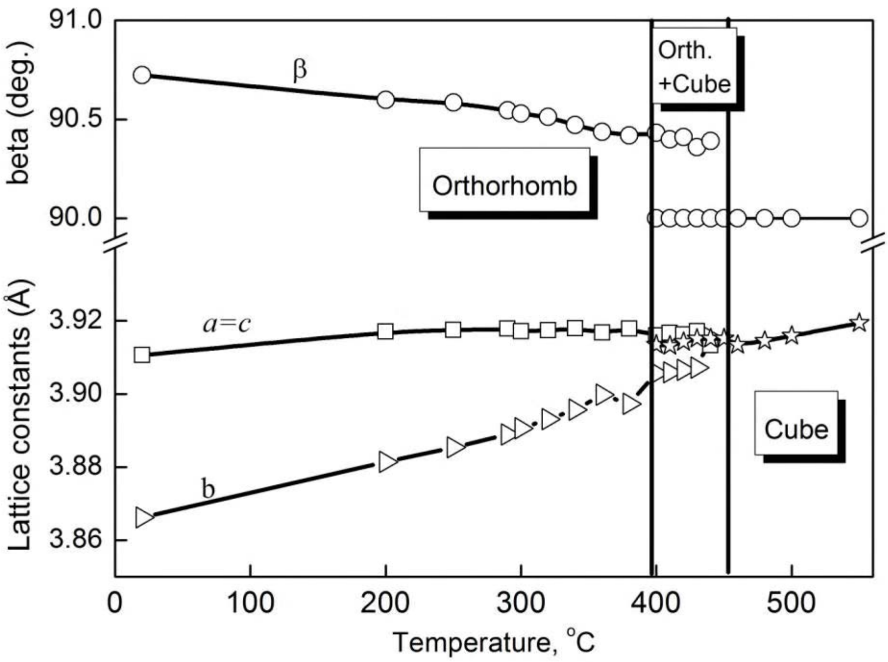

Fig 2. Lattice parameters of the annealed NLN sample in monoclinic setting.

According to both dielectric and X-ray diffraction data, heating of the samples up to 500 °C restores the initial value of phase transition temperature. As shown on the Figure 3, in restored sample an orthorhombic phase is observed in the temperature range 20-330 °C; a pure cubic phase is seen above 370 °C while in the temperature range 340-360 °C orthorhombic and cubic phases coexist (Fig.3).

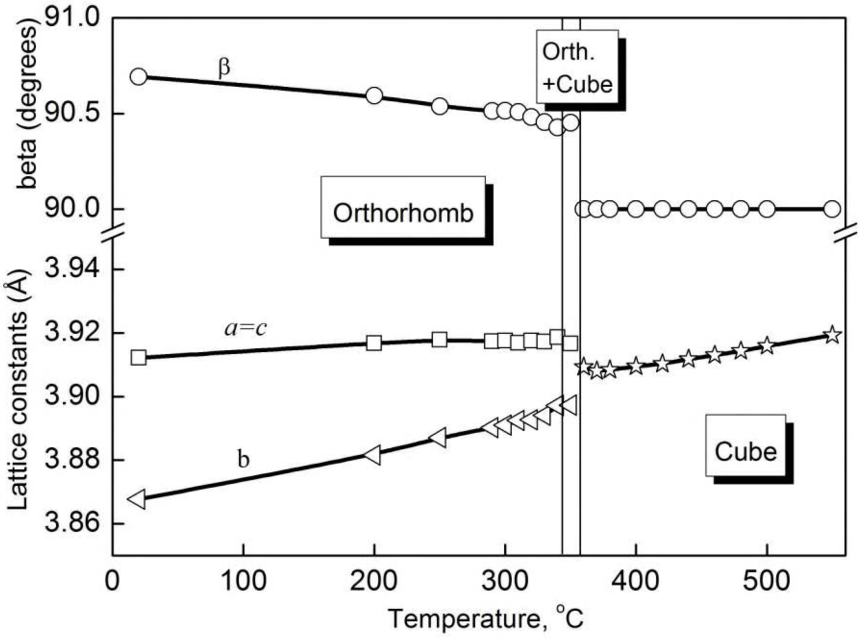

Fig 3. Lattice parameters of the annealed NLN sample after heating up to 500 °C

The increased values of T_m remain stable at least for several months providing the annealed samples are stored at room temperature.

Acknowledgement. This work was supported by the Ministry of Education and Science of the Russian Federation (project № 3.1649.2017/4.6)

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