ESRF	**Experiment title:** Revealing the average cation distribution in $Sr_{5x}Ba_{5-5x}Nb_{10}O_{30}$	**Experiment number**: HC4783
Beamline: ID22	**Date of experiment**: from: 26/01/2022 to: 31/01/2022	**Date of report**: 18/09/2023
Shifts: 15	**Local contact(s)**: Ola G. Grendal	*Received at ESRF:*

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Aim of the experiement:
The primary aim of this experiment was to determine the cation configuration, *i.e*. site occupancies of Sr^{2+} and Ba^{2+}, in $Sr_{5x}Ba_{5-5x}Nb_{10}O_{30}$ (SBN) powders made from solid-state synthesis as a function of composition and temperature. For SBN, the set of equations that constrains the site occupancies is an undertermined set of equations, thus resonant X-ray powder diffraction at ID22 was planned.

Sample description:
SBN with four different compositions were made by solid-state synthesis, and gently grinded into powders. The powders were filled in 0.5 mm diameter glass capilliaries.

Measurments conditions:
High-resolution powder diffraction was collected at ID22 at room temperature, 300, 800 and 1000 °C, at three different energies. The three energies were selected based on being far away from the Ba^{2+} K-edge (31 keV), just before and at the Ba^{2+} K-edge (37.44 and 37.47 keV).

Obtained result:
The collected data was of the needed quality, and thus Rietveld refinement could be used to obtained the site occupancies, as presented in Figure 1. Small, but significant, changes in the site occupancies was observed as a function of temperature. The most notable was the fact the the larger Ba^{2+} is found solely on the larger atomic site. Before this work, this has been the assumption in litterature, but have never been experimentally proven. Unexpectedly, the high-resolution data of ID22 also showed that for the lowest Sr-fraction studied in this work, SBN is actually orthorombic, and not tetragonal as previsoulsy belived.

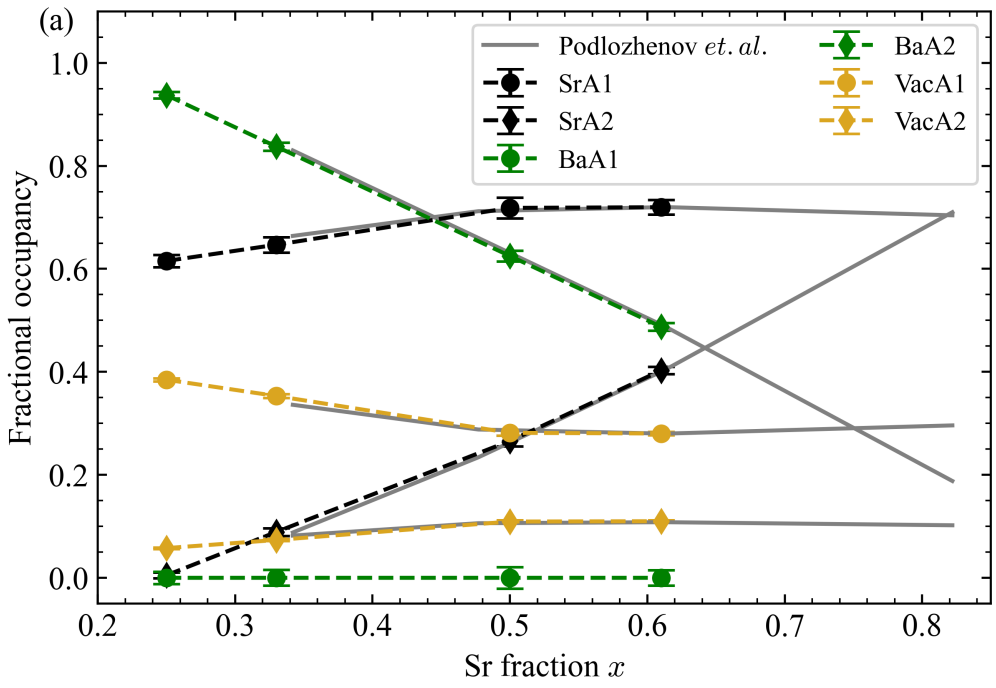

Figure 1: Fractional occupancies for Sr^{2+} and Ba^{2+} in SBN obtained from Rietveld refinement of resonant X-ray powder diffraction data.

Conclusion:
In sumary, the experiment was succsefull in determineg the site occupancies of Sr^{2+} and Ba^{2+} in SBN as a function of compositon and temperature. Thus valuble insishgt into the cation ordering in SBN was gained. This work has recently been accepted for puplication [(1)], and the abstract of this paper is included below for refference.

Refference:

(1) Grendal, O. G., Fitch, A, N., Aamlid, S. S., *A-site cation disorder in $Sr_xBa_{1-x}Nb_2O_6$ (x = 0.25, 0.33, 0.50, 0.61) studied by high-resolution resonant X-ray powder diffraction*, ACS Omega (accepted 2023).

Abstract: The dielectric and ferroelectric properties of the $Sr_xBa_{1-x}Nb_2O_6$ (SBN, $0.2 < x < 0.8$) are known to be affected by the Sr fraction, and can be further controlled by various quenching schemes. Changes in A-site cation configuration are believed to be linked to these changes in properties. In this work, we study the A-site cation disorder in SBN by the use of high-resolution resonant X-ray powder diffraction. The results show that the larger Ba^{2+} is found exclusively on the larger A2 site, while Sr^{2+} is found on both the A1 and A2 sites, with an increasing amount on A2 with increasing Sr fraction. At elevated temperatures, a small migration of Sr^{2+} from A1 to A2 is observed for SBN50 and SBN61. Linking this change in occupancies to changes in average cation size on the A1 and A2 sites allows for rationalization of the property changes observed for quenched samples. Furthermore, SBN25 is shown to deviate from the tetragonal *P*4*bm* structure, and is found to be orthorhombic with a *Cmm*2 structure.