Effects of flux treatment on morphology of single-crystalline BaNbO$_2$N particles

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Electronic Supplementary information

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(1) XRD patterns for BaNbO$_2$N treated using different alkali chloride fluxes

Figure S1 shows magnified regions of the XRD patterns in Figure 1 in the 2$\theta$ range of 30–60°. For BaNbO$_2$N(NaCl), BaNbO$_2$N(KCl) and BaNbO$_2$N(RbCl), diffraction peaks associated with NbO$_x$N$_y$ phases were also observed, indicating the chemical reduction of Nb species during the flux treatment. Figure S2 shows the 2$\theta$ range of 63–64.4°. Splitting of the (220) peak can be observed, corresponding to K$_{a1}$ and K$_{a2}$. After the flux treatment, the splitting became more pronounced, indicating an improvement in crystallinity. The dashed line indicates the (220) peak position for BaNbO$_2$N before treatment. For BaNbO$_2$N(NaCl) and BaNbO$_2$N(CsCl), the split peak shifted to higher and lower angles, respectively.

**Figure S1.** XRD patterns for BaNbO$_2$N particles (a) before and after flux treatment with (b) LiCl, (c) NaCl, (d) KCl, (e) RbCl, and (f) CsCl. Square symbols (■) indicate NbO$_x$N$_y$. 
Figure S2. XRD patterns for BaNbO$_2$N particles (a) before and after flux treatment with (b) LiCl, (c) NaCl, (d) KCl, (e) RbCl, and (f) CsCl.

(2) Schematic illustration of two-boat flux treatment setup

Figure S3 shows a schematic illustration of the two-boat flux treatment setup. Two alumina boats were inserted inside an alumina tube and were heated under an NH$_3$ flow. An NaCl flux and BaNbO$_2$N particles was placed in the upstream and downstream boats, respectively. During the flux treatment, both boats were heated to the same temperature.

Figure S3. Schematic illustration of two-boat flux treatment setup.
(3) Elemental analysis

Elemental analysis results for BaNbO$_2$N before and after flux treatment with NaCl, KCl, RbCl, and CsCl are shown in Table S1. The amount of Ba, Nb, and alkali metal (except Cs) was determined by ICP-AES. The amount of Cs was determined by XRF. The amount of O and N was determined by combustion analysis.

**Table S1.** Elemental analysis results (atomic ratio) before and after flux treatment using alkali chlorides.

<table>
<thead>
<tr>
<th>Flux</th>
<th>Cation of flux</th>
<th>Ba$^a$</th>
<th>Nb$^a$</th>
<th>O$^b$</th>
<th>N$^b$</th>
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<tr>
<td>–</td>
<td>–</td>
<td>1.00</td>
<td>0.91</td>
<td>2.66</td>
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<td>NaCl</td>
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<td>1.00</td>
<td>1.11</td>
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<tr>
<td>KCl</td>
<td>0.02$^{a,c}$</td>
<td>1.00</td>
<td>1.04</td>
<td>2.03</td>
<td>1.03</td>
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<tr>
<td>RbCl</td>
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<td>1.00</td>
<td>1.04</td>
<td>2.02</td>
<td>1.05</td>
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<tr>
<td>CsCl</td>
<td>0.03$^{c}$</td>
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<td>1.02</td>
<td>2.07</td>
<td>1.00</td>
</tr>
</tbody>
</table>

$^a$ Determined by ICP analysis  
$^b$ Determined by combustion analysis  
$^c$ Determined by XRF analysis