Effects of flux synthesis on $SrNbO_2N$ particles for photoelectrochemical water splitting

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Electronic Supplementary information

Contents

Fig. S1: Diffuse reflectance spectra of $SrNbO_2N$ particles prepared by NaCl flux method at different preparation stages.

Fig. S2: Diffuse reflectance spectra of $SrNbO_2N$ particles prepared from oxide precursors using different types of flux.

Fig. S3: SEM images of $SrNbO_2N$ particles prepared from oxide precursors using different types of flux.

Fig. S4: TEM images and SAED pattern for oxide precursor produced using NaI flux.

Fig. S5: XRD patterns, diffuse reflectance spectra, and PEC performance for oxide precursors and $SrNbO_2N$ particles prepared using NaCl, RbCl and NaI fluxes with and without pre-calcination.

Fig. S6: SEM images of pre-calcinated oxide precursors prepared using NaCl, RbCl, and NaI fluxes.

Fig. S7: SEM images of $SrNbO_2N$/Nb/Ti photoanodes. The $SrNbO_2N$ was synthesized from a pre-calcinated oxide precursor treated using NaI.

Fig. S8: Current-time curves at 1.23 V_{RHE} of $SrNbO_2N$ with different CoPi deposition time

Table S1: EDX elemental analysis results for oxide precursors treated using different types of flux.

Table S2: Elemental analysis results for oxide precursor and $SrNbO_2N$ particles treated using NaI flux.

Table S3**:** A crystal structure model of $(NaNbO_3)_{0.35}$-$(Sr_{4/3}Nb_{2/3}O_3)_{0.65}$ for Rietveld Analysis of SNO(NaI)

Table S4: Lattice constants of SNON (no flux) and SNON (AX) obtained from Rietveld Analysis.

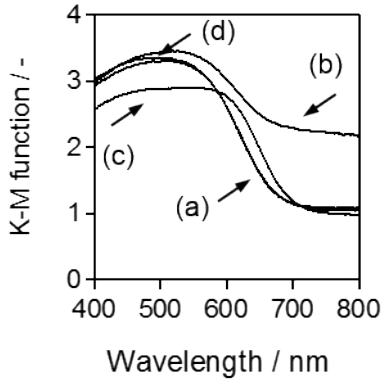

Fig. S1 Diffuse reflectance spectra of (a) SNON (no flux), (b) SNON (oxide), (c) SNON (nitridation), and (d) SNON (post).

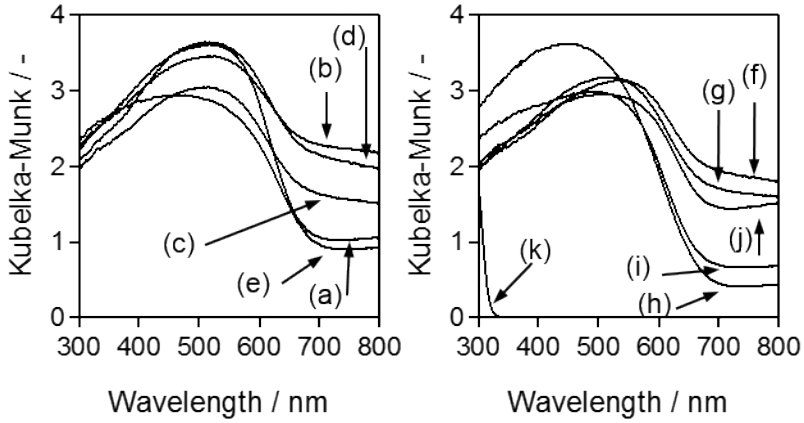

Fig. S2 Diffuse reflectance spectra of $SrNbO_2N$ particles obtained from oxide precursors prepared (a) without flux, and using (b) NaCl, (c) KCl, (d) RbCl, (e) $SrCl_2$, (f) NaBr, (g) KBr, (h) NaI, (i) KI, and (j) CsI fluxes. (k) Spectrum of oxide precursor treated prepared using NaI flux.

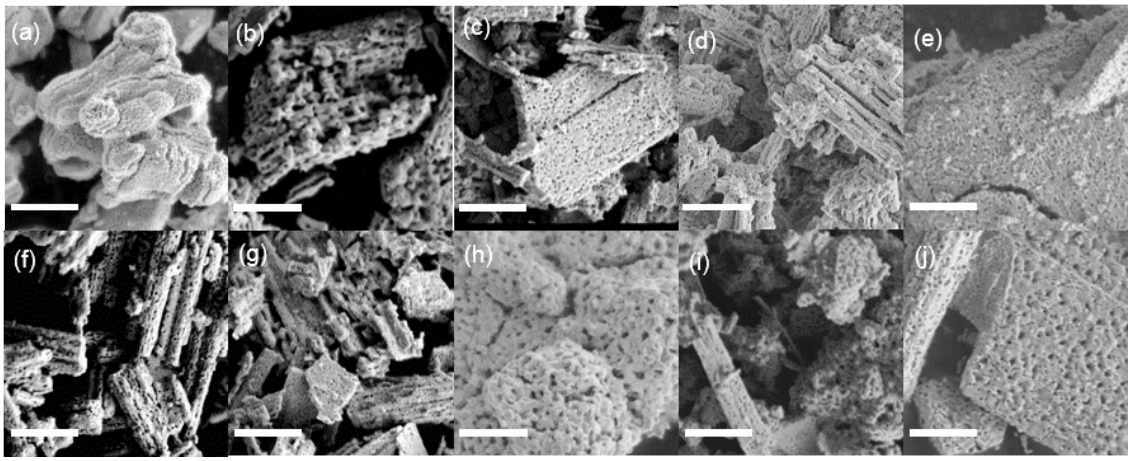

Fig. S3 SEM images of $SrNbO_2N$ obtained from oxide precursors prepared (a) without flux, and using (b) NaCl, (c) KCl, (d) RbCl, (e) $SrCl_2$, (f) NaBr, (g) KBr, (h) NaI, (i) KI, and (j) CsI fluxes. The white bars in the SEM images represent 2 μm.

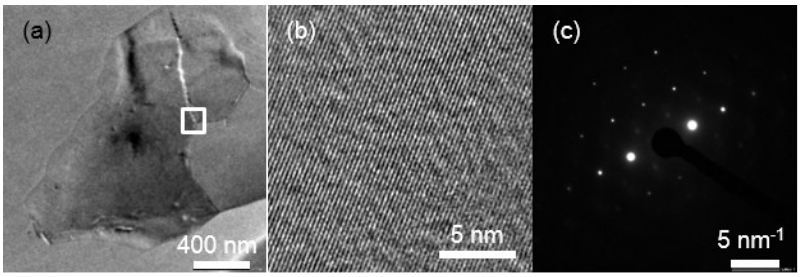

Fig. S4 (a,b) TEM images of oxide precursor prepared using NaI flux, and (c) SAED pattern from area indicated by white square in (a).

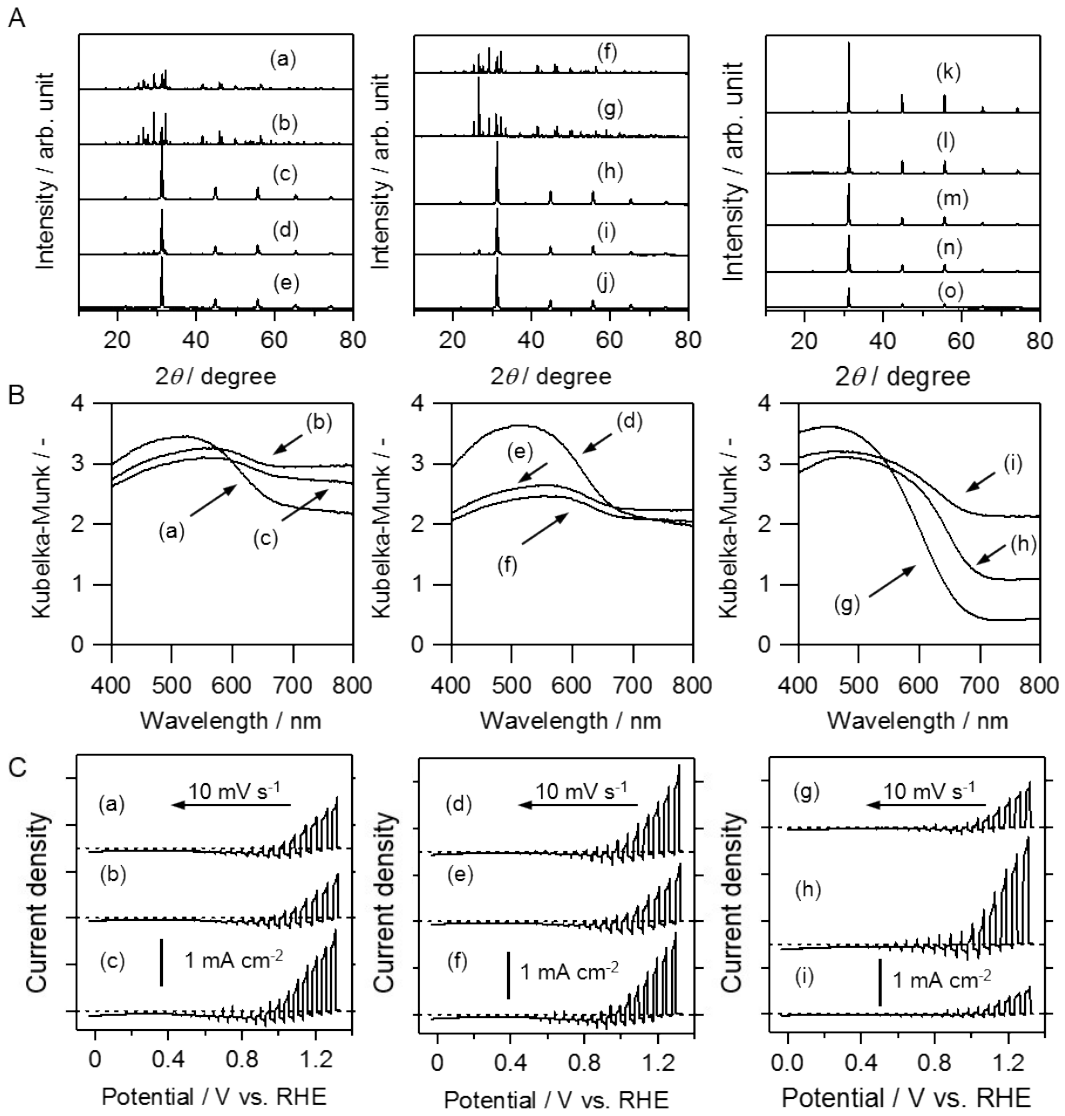

Fig. S5 (A) XRD patterns for (a) SNO (NaCl), (b) SNO (NaCl, pre-calcinated), (c) SNON (NaCl), (d) SNON (NaCl, pre-calcinated) using nitridation conditions of 1173 K for 15 h, (e) SNON (NaCl, pre-calcinated) using nitridation conditions of 1223 K for 20 h, (f) SNO (RbCl), (g) SNO (RbCl, pre-calcinated), (h) SNON (RbCl), (i) SNON (RbCl, pre-calcinated) using nitridation conditions of 1173 K for 15 h, (j) SNON (RbCl, pre-calcinated) using nitridation conditions of 1223 K for 20 h, (k) SNO (NaI), (l) SNO (NaI, pre-calcinated), (m) SNON (NaI), (n) SNON (NaI, pre-calcinated) using nitridation conditions of 1173 K for 15 h, (o) SNON (NaI, pre-calcinated) using nitridation conditions of 1223 K for 20 h, (B) Diffuse reflectance spectra and (C) current-potential curves for $SrNbO_2N$ prepared from oxide precursors using (a)-(c) NaCl flux, (d)-(f) RbCl flux, and (g)-(i) NaI flux. (a), (d), (g) without pre-calcination, (b), (e), (h) pre-calcinated and nitrided at 1173 K for 15 h, and (c),(f), (i) pre-calcinated and nitrided at 1223 K for 20 h.

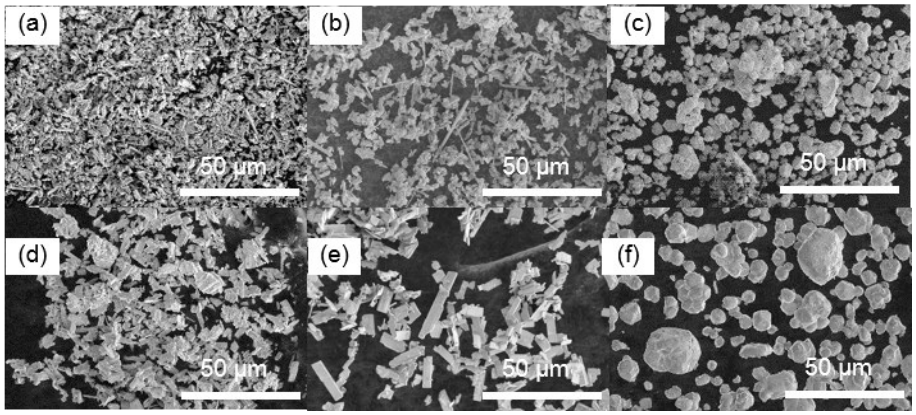

Fig. S6 SEM images of (a) SNO (NaCl), (b) SNO (RbCl), (c) SNO (NaI), (d) SNO (NaCl, pre-calcinated), (e) SNO (RbCl, pre-calcinated), and (f) SNO (NaI, pre-calcinated).

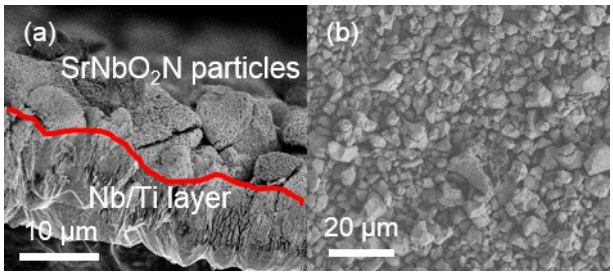

Fig. S7 SEM images of $SrNbO_2N$/Nb/Ti photoanode. $SrNbO_2N$ was synthesized from pre-calcinated oxide precursor produced using NaI flux. (a) Cross-sectional view and (b) top view of electrode.

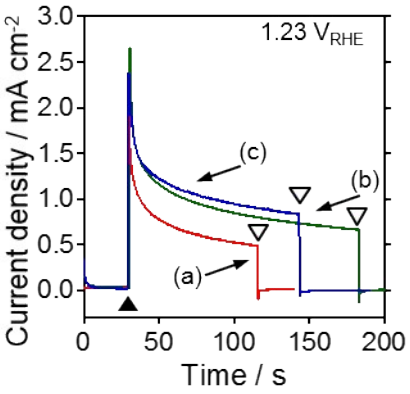

Fig. S8 Current-time curves at 1.23 V_{RHE} of $SrNbO_2N$ prepared from oxide precursors using NaI flux with different CoPi deposition time (a) 30 sec, (b) 200 sec, and (c) 1000 sec. A filled triangle indicates the time light was turned on and open triangles indicate the one was turned off.

Table S1 EDX elemental analysis results for oxide precursors prepared using different types of flux. The Nb content is normalized to 1.

Kind of flux	Cation of flux	Sr	Nb
w/o flux	-	1.01	1
NaCl	< 0.01	0.99	1
KCl	0.01	0.98	1
RbCl	0.01	1.00	1
$SrCl_2$	-	1.12	1
NaBr	< 0.01	1.00	1
KBr	0.01	0.99	1
NaI	0.35	1.13	1
KI	0.19	1.13	1
CsI	0.09	1.11	1

Table S2 Elemental analysis results for (a) oxide precursor and (b) $SrNbO_2N$ particles prepared using NaI flux.

(a)

	Molar ratio of elements / -			
	Na	Sr	Nb	O
TEM-EDX	0.36	0.87	0.78	3.00
ICP	0.39	0.87	0.83	-

(b)

	Molar ratio of elements / -			
	Na	Sr	Nb	O
TEM-EDX	0.02	1.00	1.01	-
ICP	-	1.00	0.94	-

Table S3 A crystal structure model of $(NaNbO_3)_{0.35}$-$(Sr_{4/3}Nb_{2/3}O_3)_{0.65}$ for Rietveld Analysis of SNO(NaI)

Site	Wyckoff notation*	Site occupancy (*g*)	Fractional coordinates		
			x	y	z
Na1	8c	0.03	1/4	1/4	1/4
Sr1	8c	0.97	1/4	1/4	1/4
Na2	4a	0.15	0.0	0.0	0.0
Sr2	4a	0.05	0.0	0.0	0.0
Nb1	4a	0.80	0.0	0.0	0.0
Na3	4b	0.17	1/2	1/2	1/2
Sr3	4b	0.0634	1/2	1/2	1/2
Nb3	4b	0.7666	1/2	1/2	1/2
O	24d	1.0	0.0	1/4	1/4

* K_2NaAlF_6 structure.

Table S4 Lattice constants of SNON (no flux) and SNON (AX) obtained from Rietveld Analysis.

Kind of flux	Lattice constant (nm)[a]
without	0.404946(6)
NaCl	0.404166(3)
KCl	0.404171(3)
RbCl	0.404241(3)
$SrCl_2$	0.404440(9)
NaBr	0.404211(3)
KBr	0.404180(3)
NaI	0.404620(3)
KI	0.404528(4)
CsI	0.404636(9)

[a] Values in parentheses are standard deviations in the last digits.