

Supporting information

for

Reproducible Shape Control of Single-Crystal SnO Micro Particles

*Mai Thanh Nguyen, Hiroaki Shirai, Chondanai Tiankanon, Hiroki Tsukamoto, Yohei Ishida and Tetsu Yonezawa**

Division of Materials Science and Engineering, Faculty of Engineering, Hokkaido University,
Kita 13 Nishi 8, Sapporo, Hokkaido 060-8628, Japan

Corresponding author. Email: tetsu@eng.hokudai.ac.jp

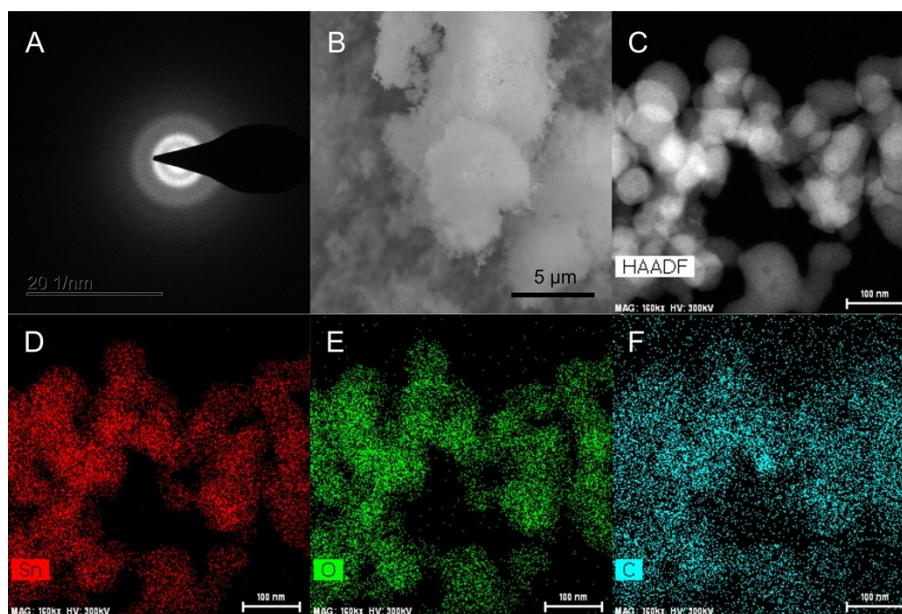


Figure S1. (A) SAED pattern, (B) SEM image, (c) HAADF image and (D-F) EDX elemental mappings for Sn L, O K, and C K of the particles synthesized using only OD at 200 °C.

Table S1. Crystallite size and strain estimation for SnO particles from the XRD peak broadening

OAM vol%	Crystallite size (nm)	Strain (%)
100	128	0.30
75	120	0.25
50	134	0.25
36	141	0.25
30	133	0.24
25	116	0.19
20	56	0.35
9	59	0.41
1	47	0.44

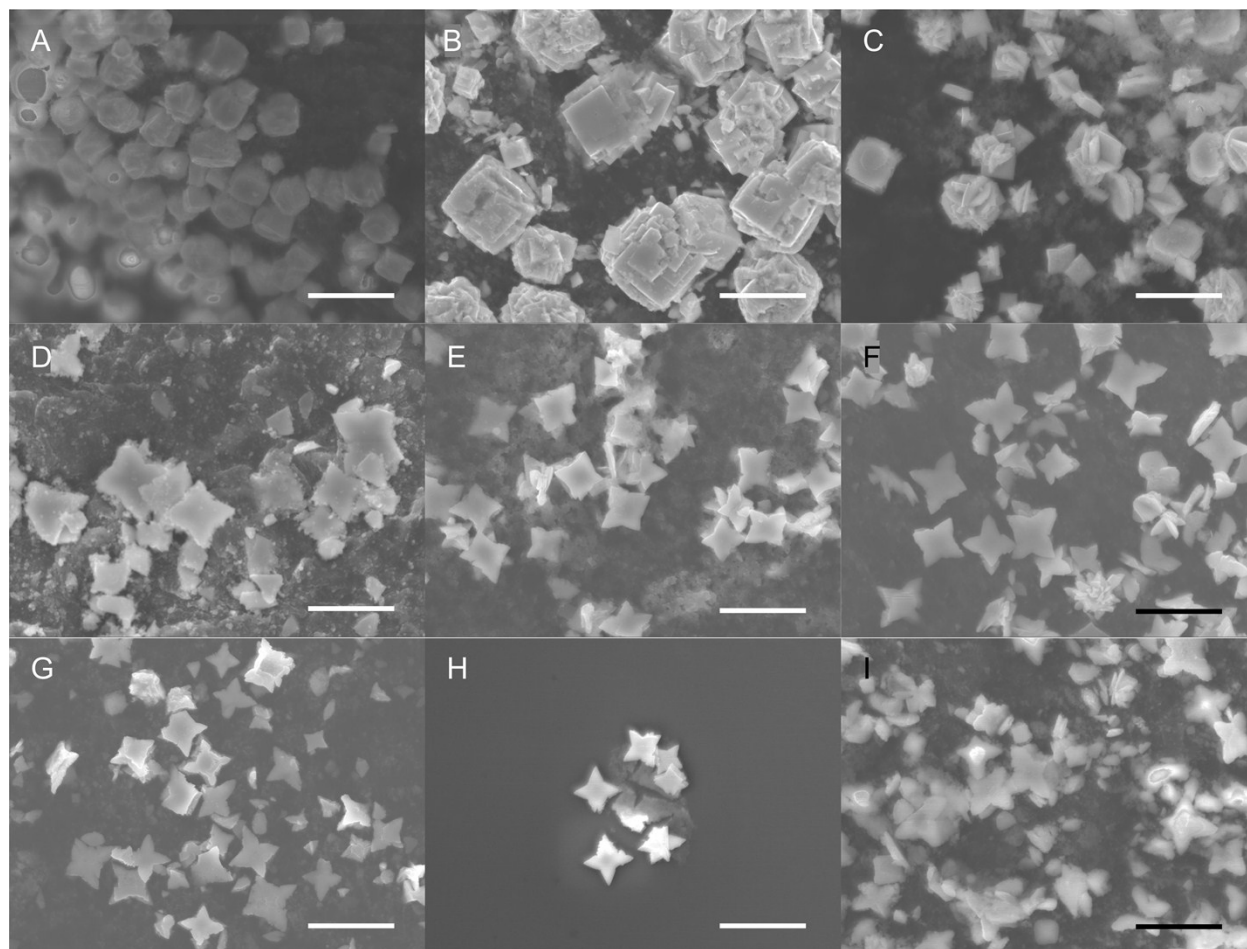


Figure S2. (A-I) SEM images of the SnO particles obtained using OAM in concentrations of 1, 9, 20, 25, 30, 36, 50, 75, and 100 vol%, respectively. All the scale bars represent 5 μm .

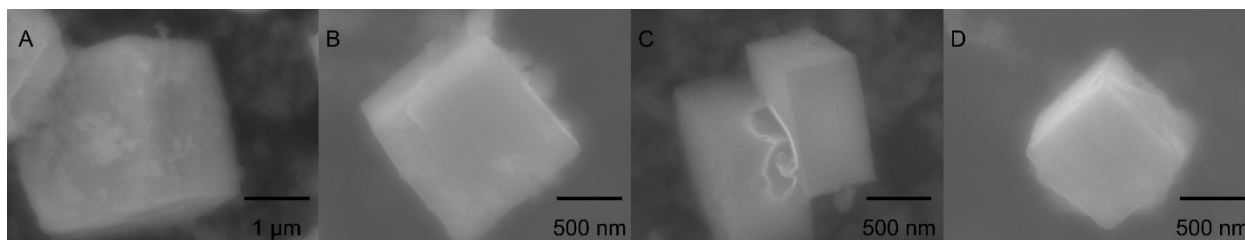


Figure S3. SEM images of SnO particles synthesized using 9 vol% OAM and same total reaction volume, and in different input amounts of Sn(acac)₂: (A) 0.76, (B, C) 0.38, and (D) 0.19 mmol. The input amount of Sn(acac)₂ of 1.52 mmol is used in the standard case shown in the main text.

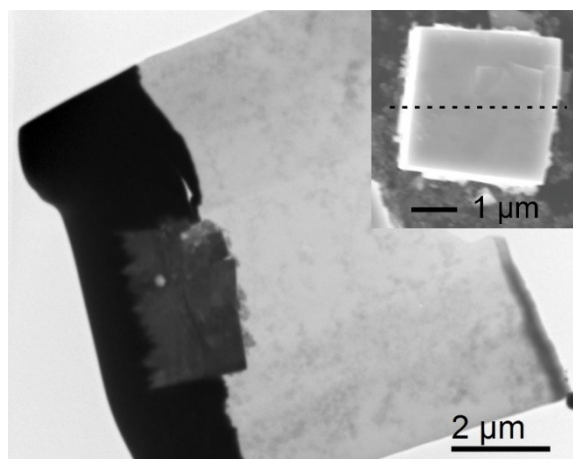


Figure S4. Cross sectional TEM image of SnO particles synthesized using 9 vol% OAM after FIB processing along the top-down direction: SnO is sandwiched between the carbon tape (grey) and the W coating layer (black); the inset shows the top view image of the cubic particle before W deposition, and the FIB cutting is along the dash line.

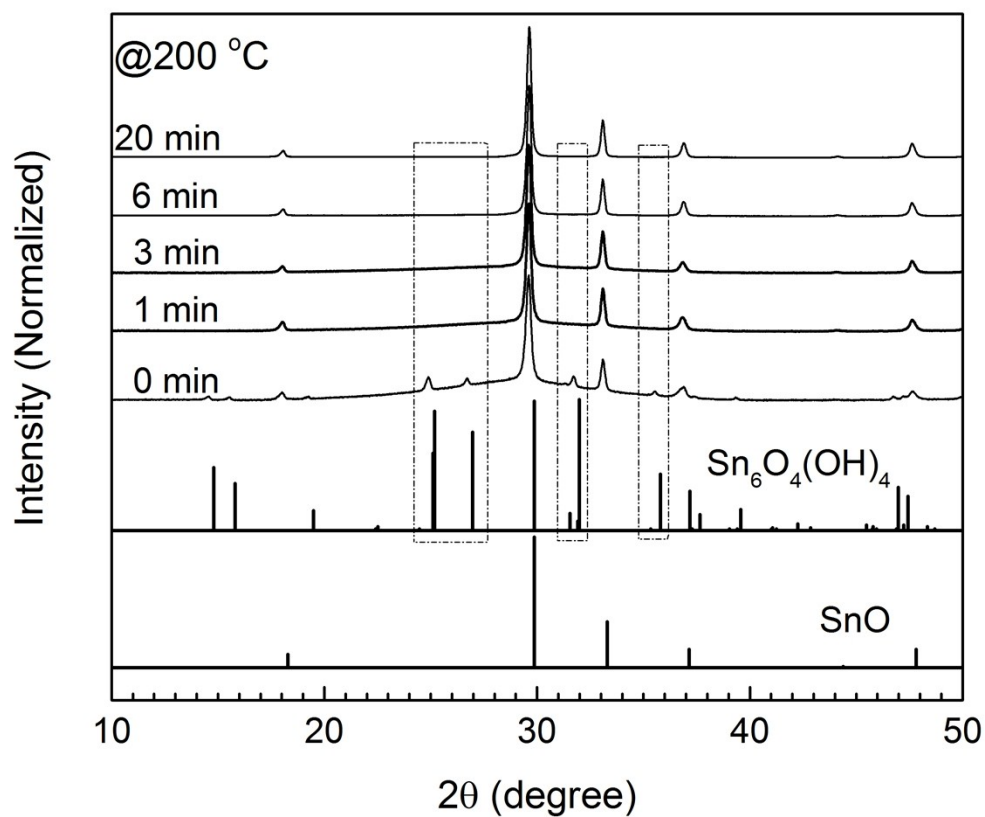


Figure S5. XRD patterns of the obtained particles synthesized using 36 vol% OAM at 200 °C after various reaction time: 0, 1, 3, 6, and 20 min. Reference patterns for tetragonal SnO (PDF No. 06-0395) and tetragonal Sn₆O₄(OH)₄ (PDF No. 046-1486) are given. The dashed rectangular windows are used for visual guide of some areas in the patterns distinguishable for only Sn₆O₄(OH)₄ peaks.

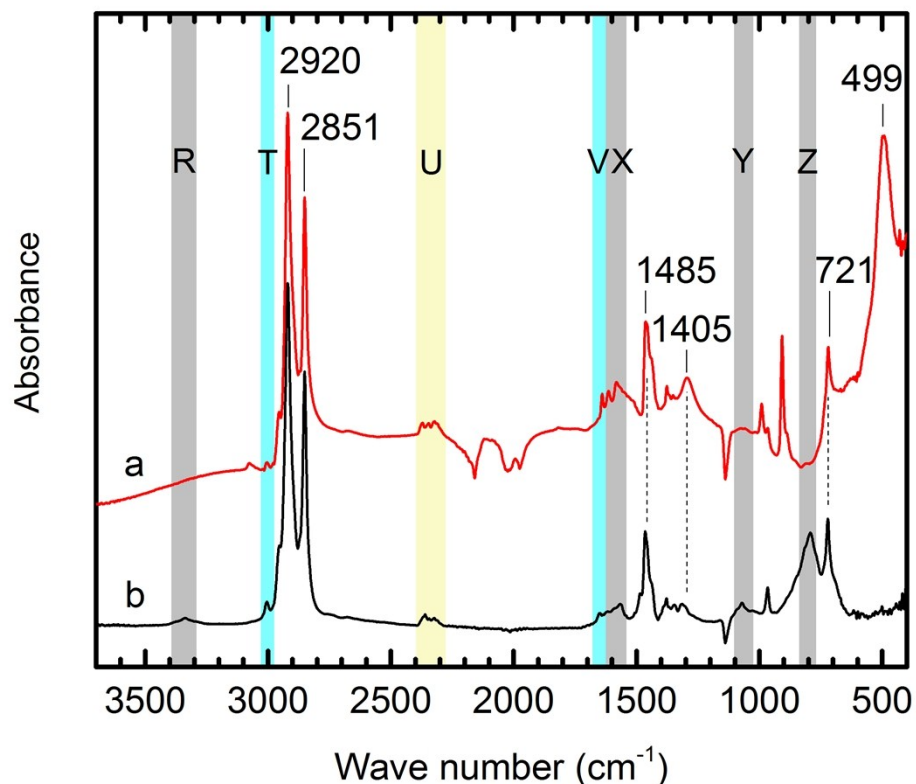


Figure S6. FT-IR spectrum of (a) purified SnO particles synthesized using 36 vol% OAM and (b) only OAM. The assignment of peaks and bands is given in Table S2.

Table S2. Infrared vibrational assignment for the FT-IR spectra in Figure S6

Peak/band ID	Frequency	Vibrational mode	SnO-OAM	Only OAM
	2920 and 2851	$\nu_{as}(\text{C-H})$ and $\nu_s(\text{C-H})$	strong, sharp	strong, sharp
	1485-1405	$\delta(-\text{CH}_3)$	medium	strong-weak
	721	$\delta(\text{C-C})$	medium	medium
	499	$\nu(\text{Sn-O})$	strong	none
R	3300-3360	$\nu_{as}(\text{NH}_2)$ and $\nu_s(\text{NH}_2)$	very weak	weak
X	1550-1620	$\delta(\text{NH}_2)$	medium	medium
Z	790-810	$\delta(\text{NH}_2)$	weak	strong
Y	1060-1090	$\delta(\text{C-N})$ in amine	weak	medium
T	3003-3006	$\nu(\text{=C-H})$	weak	medium
V	1640-1660	$\nu(\text{-C=C-})$	medium	medium
U*	2320-2400	$\nu(\text{O=C=O})$	From the environment	

ν , ν_{as} , ν_s , δ are stretching, as-symmetric stretching, symmetric stretching and bending vibrations, respectively.

The peaks located between Y and Z bands may belong to the stretching vibration of trans(-CH=). A band at 1000 cm⁻¹ is an un-assigned band, this presents in FT-IR spectra of all SnO samples.

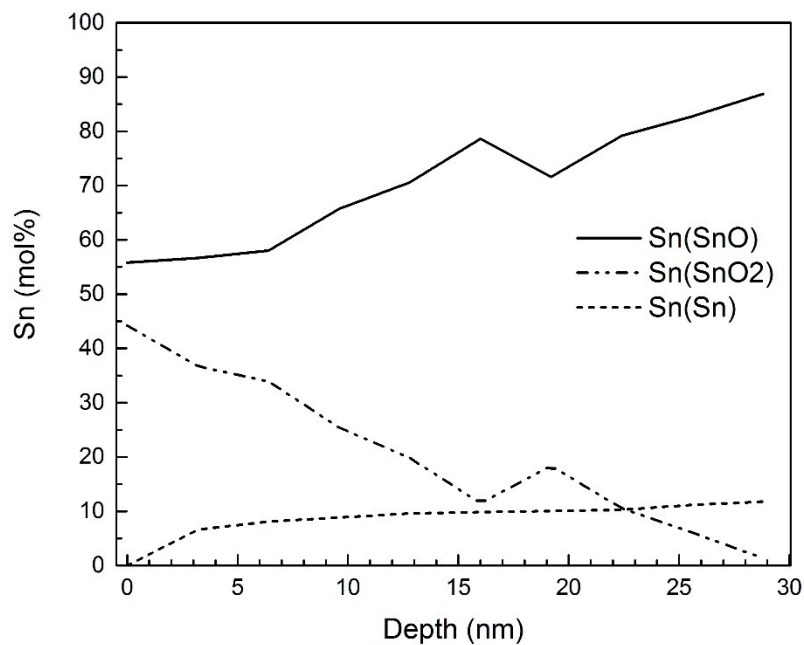


Figure S7. AES depth profile of SnO particles synthesized using 30 vol% OAM. Depth profile was collected using Ar etching with etching rate of 3.2 nm/min. The mol% of Sn in SnO, SnO₂, and metallic Sn were plotted using solid, dashed, and dash-dot-dot curves, respectively.

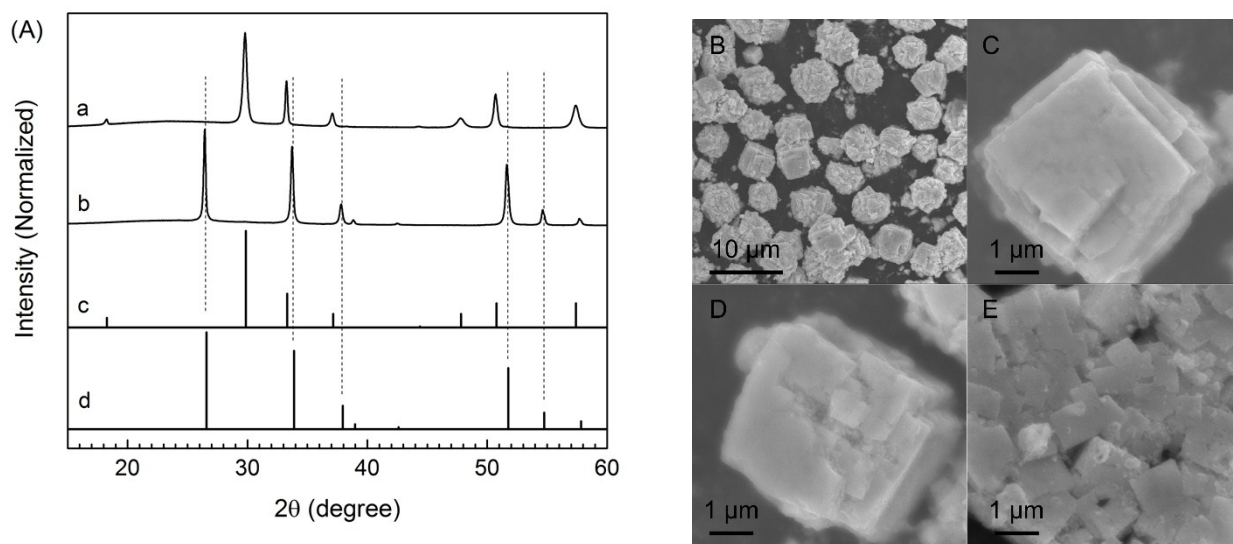


Figure S8. (A) XRD patterns of (a) the as-synthesized SnO particles using 9 vol% OAM, (b) resulting samples after annealing in the air at 700 °C for 2 h, and reference patterns for (c) tetragonal SnO (PDF No. 06-0395), and (d) tetragonal SnO₂ (PDF No. 021-1250) respectively. (B-D) Corresponding SEM images of the annealed particles. The dash lines, which marked some main peak positions of tetragonal SnO₂ were used to guide the eyes.

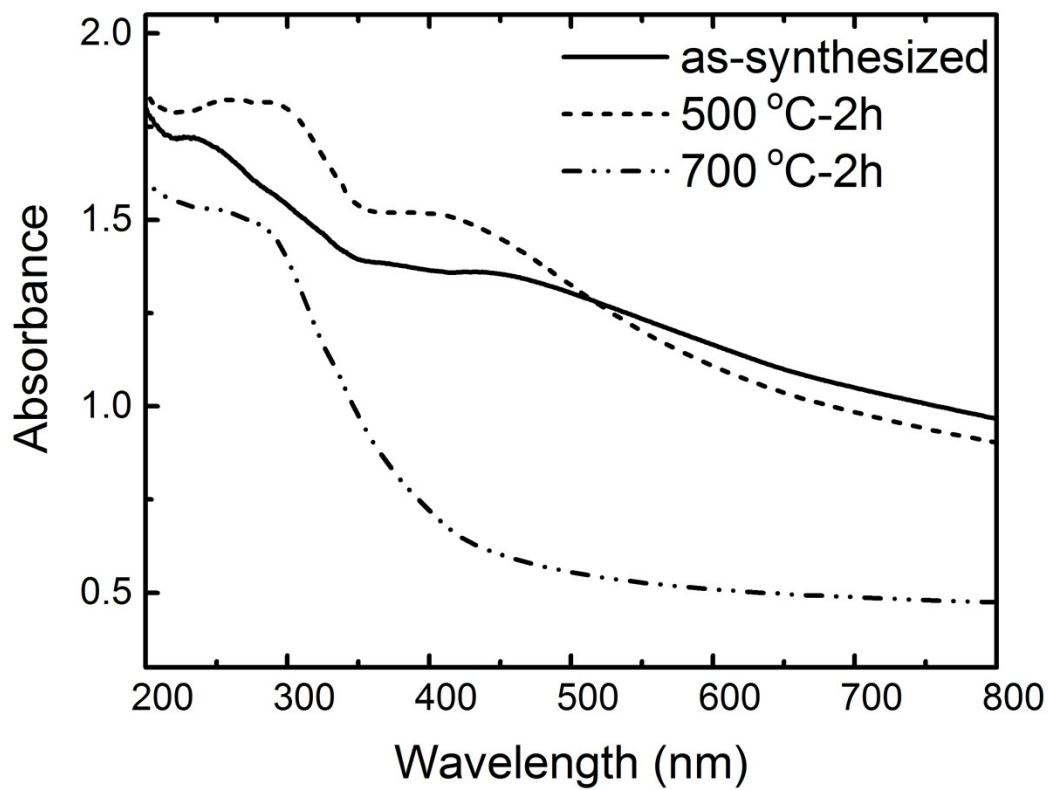


Figure S9. Absorption spectrum of the SnO particles synthesized using 75 vol% OAM at room temperature (solid curve) and those obtained after annealing at 500 °C (dash curve) and 700 °C for 2 h (dash-dot-dot curve).