Supplementary Information forw

Rational design of the fabrication of bulk Ni_3Sn_2 alloy catalysts for the synthesis of 1,4-pentanediol from biomass-derived furfural without acidic co-catalyst

Rodiansono,*[a,b] Atina Sabila Azzahra,[b] Pathur Razi Ansyah,[c] Sadang Husain,[d] and Shogo Shimazu[e]

[a]Department of Chemistry, Lambung Mangkurat University, Jl. A. Yani Km 36 Banjarbaru, Indonesia 70714
[b]Inorganic Materials & Catalysis Laboratory (IMCat), Catalysis for Sustainable Energy and Environment (CATSuRe), Lambung Mangkurat University.
[c]*Department of Physics, Lambung Mangkurat University, Jl. A. Yani Km 36 Banjarbaru, Indonesia 70714.*
[d]*Department of Mechanical Engineering, Lambung Mangkurat University, Jl. A. Yani Km 35 Banjarbaru, Indonesia 70714.*
[e]Graduate School of Engineering, Chiba University, 1-33 Yayoi, Inage-ku, Chiba, Japan 263-8522
*Corresponding author: rodiansono@ulm.ac.id (R. Rodiansono); Tel./fax.: +625114773112

Contents

1. Results of catalyst characterisation

Table S1. Specific surface area BET (S_{BET}) and average crystallite sizes of Ni_3Sn_2(101) alloy phase of bulk Ni-Sn(1.5) alloy synthesized at different parameters

Entry	Catalyst[a]	S_{BET}[b] (m^2/g)	D[c] (nm)
1	Ni_3Sn_2 (2-Me-EtOH; pH = 8; 573 K/H_2)	9.7	14.0
2	Ni_3Sn_2 (2-Me-EtOH; pH = 8; 673 K/H_2)	70.3	17.0
3	Ni_3Sn_2 (2-Me-EtOH; pH = 8; 773 K/H_2)	1.7	26.5
4	Ni_3Sn_2 (2-Me-EtOH; pH = 8; 873 K/H_2)	2.9	28.4
5	Ni_3Sn_2 (2-Me-EtOH; pH = 8; HT 12 h; 673 K/H_2)	21.6	13.7
6	Ni_3Sn_2 (2-Me-EtOH; pH = 8; HT 48 h & 673 K/H_2)	10.6	19.4
7	Ni_3Sn_2 (2-Me-EtOH; pH = 8; HT 72 h & 673 K/H_2)	31.4	16.8
8	Ni_3Sn_2 (EG; pH = 8; 673 K/H_2)	12.0	16.5
9	Ni_3Sn_2 (without polyol; 673 K/H_2)	6.92	17.4

[a]The samples Ni_3Sn_2 samples were synthesized at different parameters, unless otherwise stated. [b] Specific surface are BET (S_{BET}) was derived from N_2 physisorption at 77 K. [c] Average crystallite sizes of Ni_3Sn_2 (101) alloy phase at 2θ =30.1° was calculated by using Scherrer`s equation.

Table S2. Identified Ni–Sn alloy phases from bulk Ni-Sn(1.5) after reduction with H_2 at 673-873 K for 1.5 h[a]

Temperature of H_2 reduction (K)		
673	**773**	**873**
92% Ni_3Sn_2 alloy phase[c] Ni_3Sn_2(101), Ni_3Sn_2(002), Ni_3Sn_2(102), Ni_3Sn_2(110), Ni_3Sn_2(201), Ni_3Sn_2(112), Ni_3Sn_2(103), Ni_3Sn_2(202), Ni_3Sn(002),	89% Ni_3Sn_2 alloy phase[c] Ni_3Sn_2(101), Ni_3Sn_2(002), Ni_3Sn_2(102), Ni_3Sn_2(110), Ni_3Sn_2(201), Ni_3Sn_2(112), Ni_3Sn_2(103), Ni_3Sn_2(202), Ni_3Sn(002),	Ni_3Sn_2(101),Ni_3Sn_2(002), Ni_3Sn_2(102) Ni_3Sn_2(110),Ni_3Sn_2(201), Ni_3Sn_2(112), Ni_3Sn_2(103), Ni_3Sn_2(202),Ni_3Sn(002),

The samples Ni-Sn(1.5) were synthesized at pH adjustment of 8, temperature hydrothermal of 423 K for 24 h, unless otherwise stated. [a]Based on the JCPDS-ISSD card of the existing Ni-Sn alloys and JCPDS card number of #06-414 for Ni_3Sn_2 [1,2]. [b]Values in the parenthesis indicate the Ni/Sn molar ratio. [c]The Mol% of alloy component was calculated by Multi-Rietveld Analysis Program LH-Riet 7.00 method on the Rietica software.

Table S3 Calculated lattice parameters (d/nm) of Ni_3Sn_2 alloy obtained at different pH of Ni-Sn solution. The data referred to the XRD patterns in Fig. 7 (in the text) at 2θ = ~44.2 (Ni_3Sn_2 (110) phase.

Entry	Sample	*d* [a](nm)
1	Ni_3Sn_2 (2-Me-EtOH; pH = 6; 673 K/H_2)	0,2057
2	Ni_3Sn_2 (2-Me-EtOH; pH = 8; 673 K/H_2)	0,2057
3	Ni_3Sn_2 (2-Me-EtOH; pH = 10; 673 K/H_2)	0,2055
4	Ni_3Sn_2 (2-Me-EtOH; pH = 12; 673 K/H_2)	0,2054

[a]The basal spacing was calculated using Bragg`s equation at at 2θ = ~44.2 (Ni_3Sn_2 (110) phase.

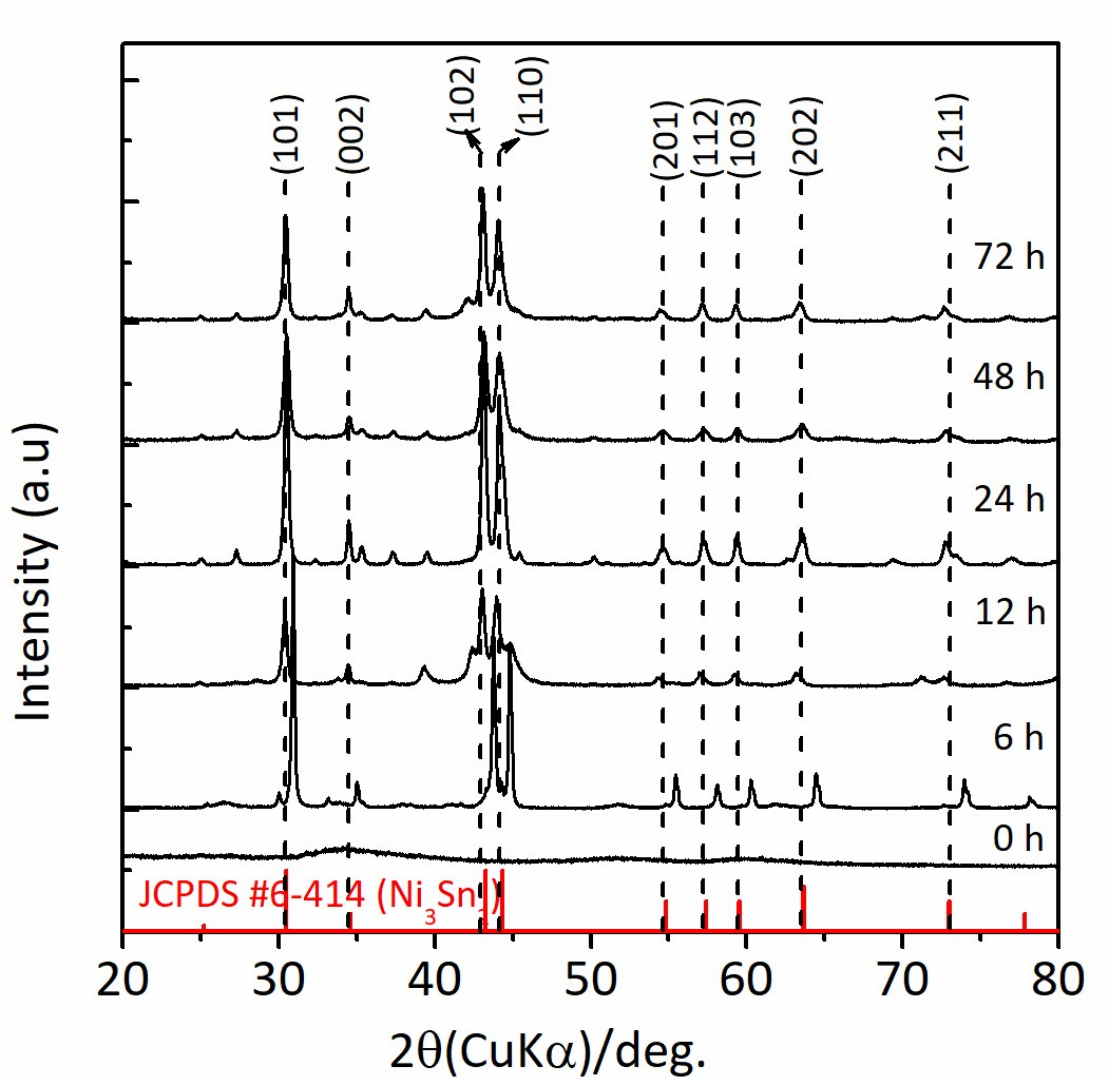

Figure S1. XRD patterns of the bulk Ni-Sn(1.5) synthesized at different hydrothermal time of 0-72 h at 423 K after reduction with H_2 at 673 K for 1.5 h.

Table S4 Calculated lattice parameters (d/nm) of Ni_3Sn_2 alloy obtained at different hydrothermal time. The data referred to the XRD patterns in Fig. S1 at 2θ = ~44.2 (Ni_3Sn_2 (110) phase.

Entry	Sample	*d* [a](nm)
1	Ni_3Sn_2 (2-Me-EtOH; pH = 8; 12 h; 673 K/H_2)	0,2061
2	Ni_3Sn_2 (2-Me-EtOH; pH = 8; 24 h; 673 K/H_2)	0,2054
3	Ni_3Sn_2 (2-Me-EtOH; pH = 8; 48 h; 673 K/H_2)	0,2053
4	Ni_3Sn_2 (2-Me-EtOH; pH = 8; 72 h; 673 K/H_2)	0,2055

[a]The basal spacing was calculated using Bragg`s equation at 2θ = ~44.2 (Ni_3Sn_2 (110) phase.

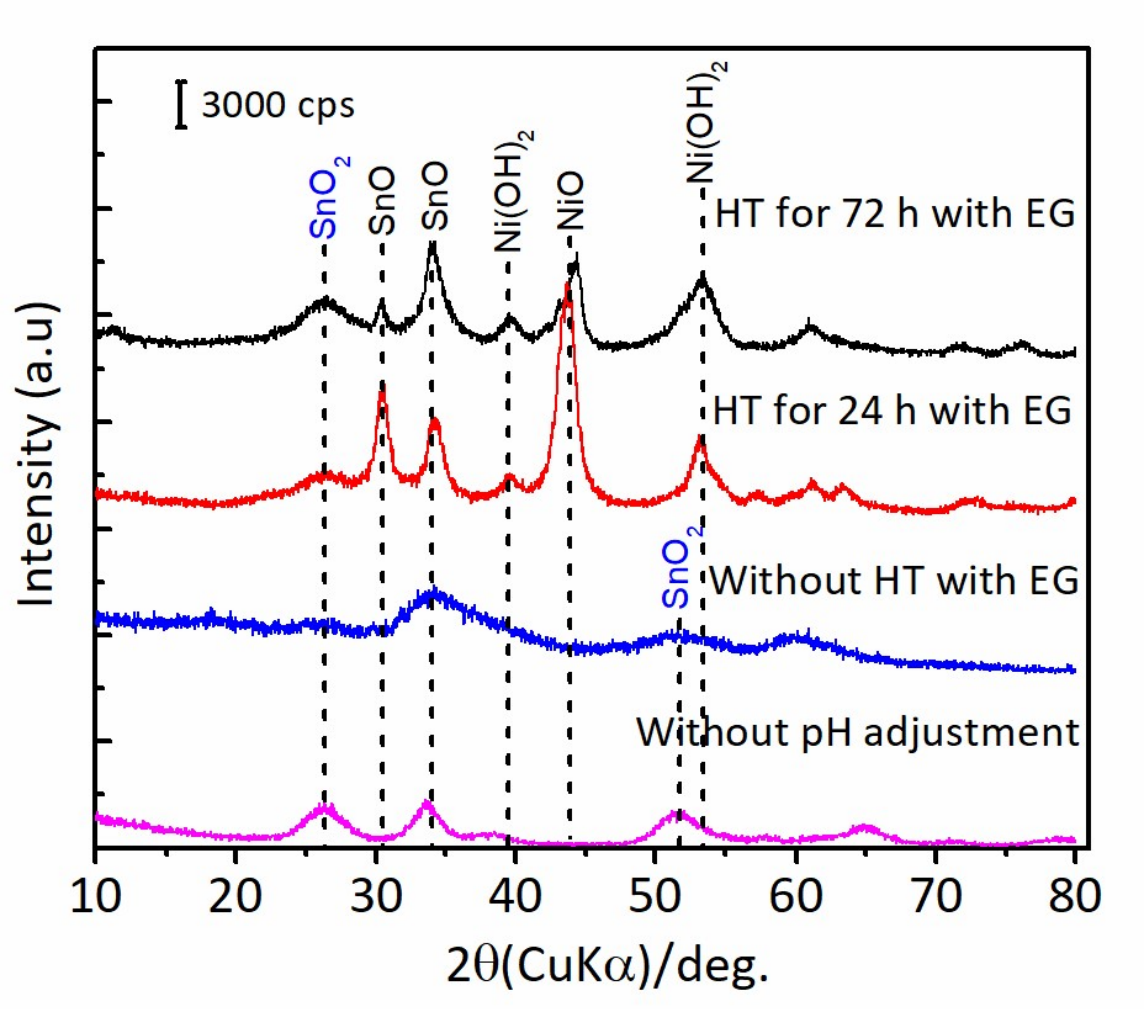

Figure S2. XRD patterns of the as-prepared bulk Ni-Sn(1.5) with different conditions.

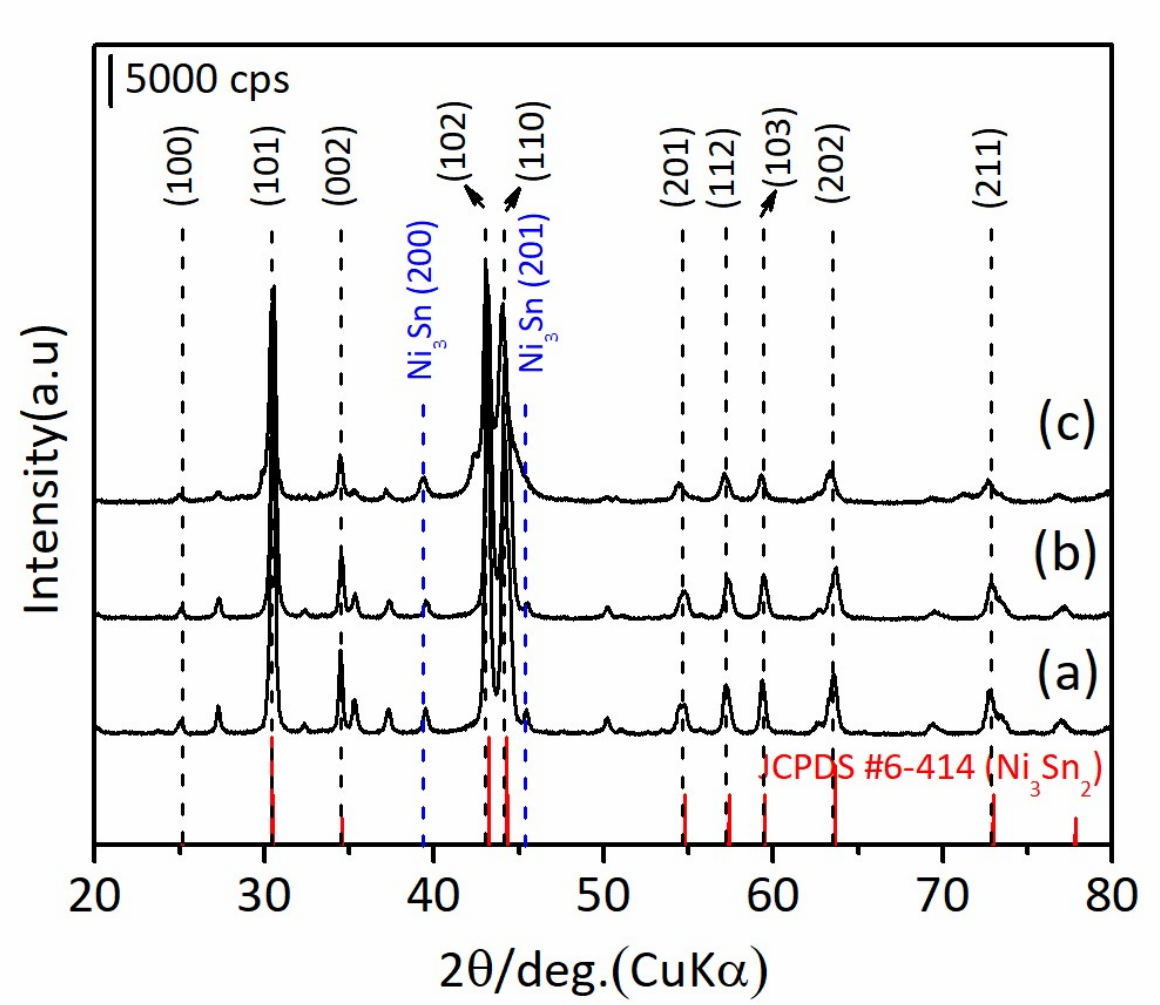

Figure S3. XRD patterns of the bulk Ni-Sn(1.5) synthesized in the presence of polyols (a) ethylene glycol (EG), (b) 2-methoxyethanol, and (c) without the addition of polyols during the hydrothermal at 423 K for 24 h, followed by reduction with H_2 at 673 K for 1.5 h.

Table S5 calculated lattice parameter (*d* (nm) of Ni_3Sn_2 samples obtained at the effect of EG, methoxy ethanol, and without polyol additives. The data referred to the XRD patterns in Fig. S3 at 2θ = ~44.2 (Ni_3Sn_2 (110) phase.

Entry	Sample	d^a (nm)
1	Ni_3Sn_2 (without polyol; 673 K/H_2)	0,2056
2	Ni_3Sn_2 (2-Me-EtOH; pH = 8; 673 K/H_2)	0,2054
3	Ni_3Sn_2 (EG; pH = 8; 673 K/H_2)	0,2050

[a]The basal spacing was calculated using Bragg`s equation at 2θ = ~44.2 (Ni_3Sn_2 (110) phase.

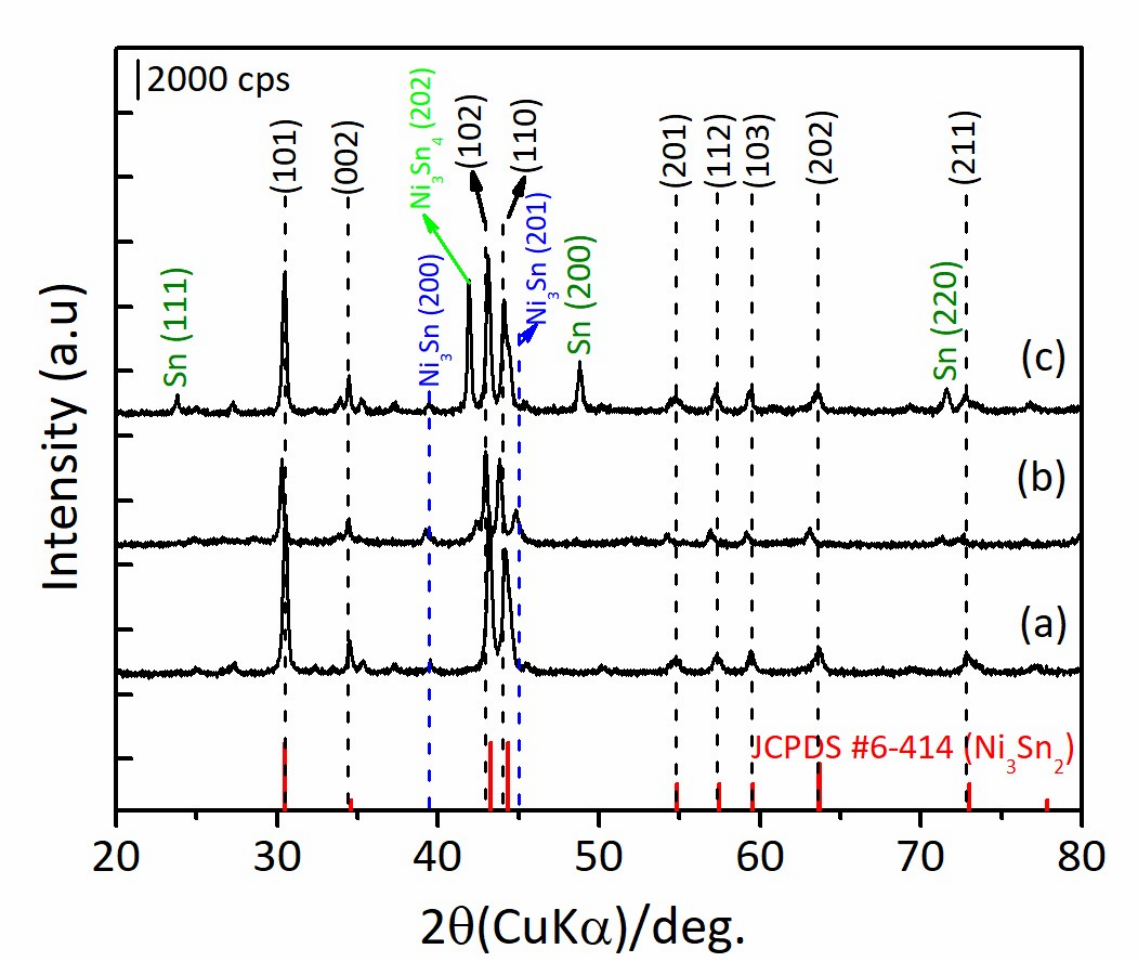

Figure S4. XRD patterns of the bulk Ni-Sn(1.5) synthesized at different temperature of hydrothermal (a) 423 K, (b) 473 K, and (c) 523 K for 24 h followed by reduction with H_2 at 673 K for 1.5 h.

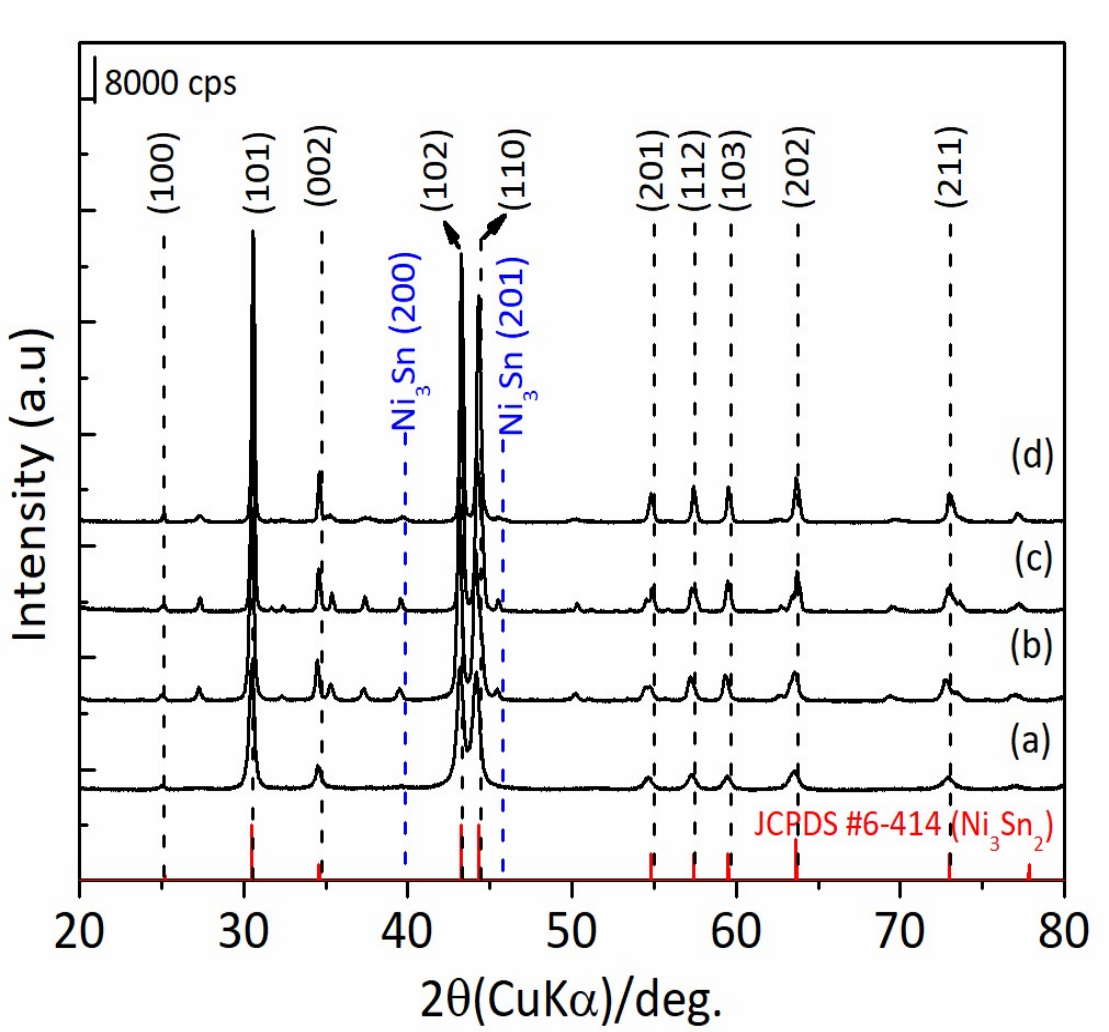

Figure S5. XRD patterns of bulk Ni-Sn(1.5) after reduction with H_2 at (a) 573 K, (b) 673 K, (c) 773 K, and (d) 873 K for 1.5 h. The plotted data were compared with JCPDS card of #06-414 (Ni_3Sn_2).

Table S6 Calculated lattice parameters (d/nm) of Ni_3Sn_2 alloy obtained at reduction temperature. The data referred to the XRD patterns in Fig. S5 at 2θ = ~44.2 (Ni_3Sn_2 (110) phase.

Entry	Sample	*d* [a](nm)
1	Ni_3Sn_2 (2-Me-EtOH; pH = 8; 24 h; 573 K/H_2)	0,2049
2	Ni_3Sn_2 (2-Me-EtOH; pH = 8; 24 h; 673 K/H_2)	0,2054
3	Ni_3Sn_2 (2-Me-EtOH; pH = 8; 24 h; 773 K/H_2)	0,2051
4	Ni_3Sn_2 (2-Me-EtOH; pH = 8; 24 h; 873 K/H_2)	0,2045

[a]The basal spacing was calculated using Bragg`s equation at 2θ = ~44.2 (Ni_3Sn_2 (110) phase.

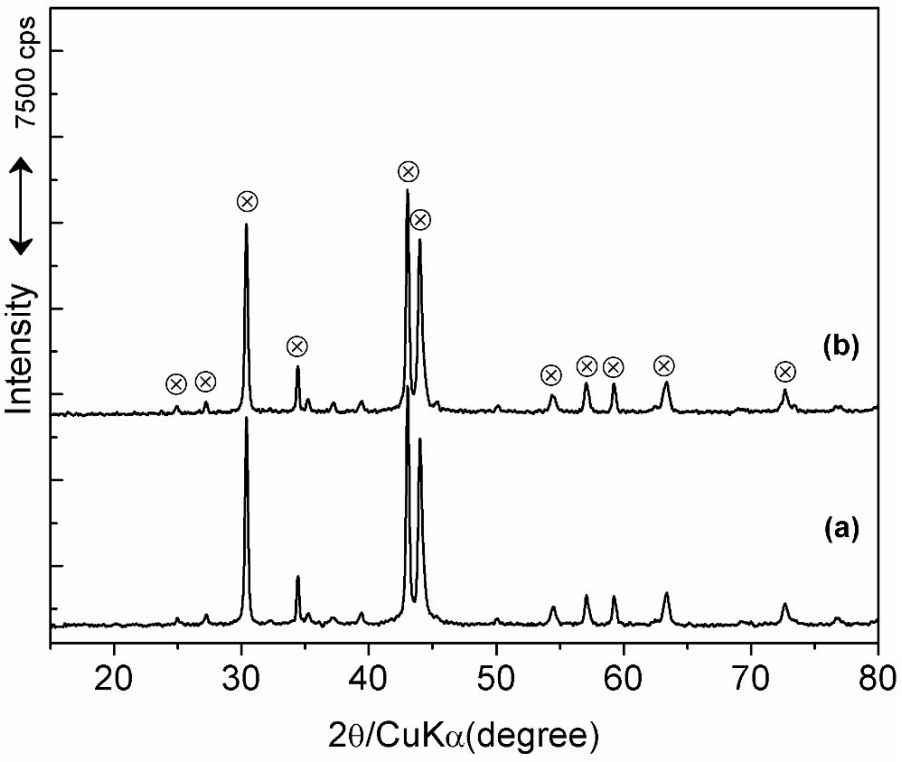

Figure S6. XRD patterns of (a) fresh Ni_3Sn_2 pH = 8 and (b) recovered after the fourth reaction run in batch reactor system after reduction with H_2 at 673 K for 1 h. ⊗ Ni_3Sn_2 alloy phase, plotted data were compared with JCPDS card of #06-414 (Ni_3Sn_2).