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研究課題名(和文) Development of a chemical synthesis technique to prepare Cu-Co NWS

研究課題名(英文) Development of a chemical synthesis technique to prepare Cu-Co NWS

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研究成果の概要(和文)：Cu-Coナノチューブとナノ粒子はアルコール還元法により合成され、生成メカニズムを提案した。溶媒(アルキル鎖が長いアルコール)、界面活性剤、金属源などは、化学的計算を用いて選択し、実験的に検証された。この点で、1-ヘプタノールは還元電位の低いCu-Coの生成に適していた。同様に、オレイルアミンとCl-/O<sub>2</sub>の比率はNTsの形成に重要な役割を果たしますが、N<sub>2</sub>の流量と金属酢酸塩/金属塩化物の混合物がCl-/O<sub>2</sub>の比率を制御します。Cu-Co NTは、CO<sub>2</sub>からギ酸へのファラデー効率がCu-Co NPのそれよりも4倍高かった。NTの高い電気触媒特性は、表面における高濃度のCo酸化物の存在による。

研究成果の学術的意義や社会的意義

(1) Cuベースの1次元NSの合成、それらの形成メカニズム、および寸法、組成を制御するパラメーターは、計算化学を使用して理論的に研究され、後で実験的に検証された。特に、還元剤と界面活性剤の選択は、計算化学から得られた結果に基づいて行われました。そして、合成実験は理論的結果に基づいて設計されました。2) この研究で開発された材料は、さらに改善を施せば、非汚染物質の生成物とエネルギーを生成することにより空気品質を向上させるためのCO<sub>2</sub>還元反応器の電極としての用途に検討できます。

研究成果の概要(英文)：Cu-Co nanotubes (NTs) and nanoparticles (NPs) have been successfully synthesized through alcohol reduction method and a mechanism formation was proposed. Parameters such as solvent, surfactant and metallic sources have been studied experimentally and chemical calculations. Theoretically, alcohols with long alkyl chain can reduced metals with low reduction potential such as Cu and Co. In this regard, 1-heptanol was suitable to produce Cu-Co NTs and NPs. Likewise, although oleylamine and the ratio Cl-/O<sub>2</sub> play important role in the formation of NTS, the N<sub>2</sub> flow rate and the mixture of metallic acetate/metallic chloride control the ratio Cl-/O<sub>2</sub>. The Cu-Co NTs exhibit a CO<sub>2</sub>-to-formate faradaic efficiency was four times higher than that of Cu-Co NPs. The high electrocatalytic properties of NTs was due to the presence of high Co oxide content on Cu-Co NT surface.

研究分野：Nanomaterials

キーワード：Alcohol Reduction 1-heptanol Oleylamine Cu-Co CO<sub>2</sub> reduction reaction Nanoparticles Nano wires Nanotubes

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様式 C - 19、F - 19 - 1、Z - 19 (共通)

## 1. 研究開始当初の背景

Multi-metallic nanostructures (NSs) are widely studied for their potential use in multiple fields such as electronic, medicine, optics, sensors, and magnetism. In this regard, multi-metallic NSs with well-defined size, shape, composition, and metallic distribution are required with the aim of tuning their physical and chemical properties through the modification of the electronic state of individual metallic elements.

On the other hand, there is a great concern about the limited source material for generating energy and an elevated increase of CO and CO<sub>2</sub> levels in the atmosphere due to use carbon-based energetic materials. So, researches have focused in the transformation of CO and CO<sub>2</sub> on useful compounds and create energy from these gases. In this case, Cu based nanoparticles (NPs) have been examined as an electrode in the CO reduction to produce acetate and ethylene. A strong synergetic effect of Cu on Co catalyst has also been observed in the gas conversion reactions to produce alcohols. Likewise, 1-D Cu-Co nanostructures (NSs) are investigated in the energy production due to their high surface area and the electron transport behavior. The catalytic and selective performance could be enhanced by using homogeneously distributed Cu and Co metals in nanowires (NWs). Still, the synthesis of 1-D Cu-Co NSs resulted in being complicated, hard to repeat and also lacks technically in dimension and composition control.

Long alkyl alcoholic technique has been developed by our group to prepare metallic NPs with controllable dimensions, shape and compositions because alcohols exhibit two dominant roles during the synthesis of metallic nanomaterials. First, the weak reducing ability that eases the control of nucleation and growth of NPs. Secondly, the long alkyl chains that avoid the agglomeration and growth. As a consequence, alcohol synthesis technique could be suitable for synthesizing Co-Cu NSs.

## 2. 研究の目的

Preparation of Cu-Co NS electrodes suitable for the formation of formic acid via electrochemical conversion of CO<sub>2</sub>. The research areas are as follows: **(a)** examination of the reducing ability of alcohols **(b)** reduction and growth mechanism of Cu-Co NSs by using alcohols, **(c)** control of composition and physical dimensions and **(d)** evaluation of catalytic properties of Cu-Co NWs.

## 3. 研究の方法

Reduction potentials of alcohols from methanol to 1-octanol were determined by calculation chemistry using Gaussian 09, Rev. E.01. Cu-Co NSs were synthesized as follows: specific concentrations of CuCl<sub>2</sub>, anhydrous, Co(OAc)<sub>2</sub>·4H<sub>2</sub>O and oleylamine were dissolved in 100 mL of 1-heptanol and then heated to 171 °C for 4.5 h under N<sub>2</sub> atmosphere. The electrochemical reduction of CO<sub>2</sub> to formic acid using Cu-Co NSs as electrodes was examined through three-electrode electrochemical cell using NHCO<sub>3</sub> as electrolyte.

## 4. 研究成果

The synthesis of high quality of Cu-Co NSs depends on (a) reducing ability of alcohols, (b) the shape of metallic seeds and (c) the adsorption of surfactant. Here, these three factors are examined to obtain a high yield of Cu-Co NWs by using alcohol as a reducing agent.

**(a) Reducing ability of alcohols:** Different reducing agents and solvents have been studied in the synthesis of Cu-based NWs; however, their potentials as reducing agents have been limited due to the poor understanding of the redox processes. Here, the molecular orbital calculation has been used to evaluate the reduction potential of alcohols through the electron transfer ability between alcohol and metal by estimating the HOMO energy of alcohol and LUMO energy of metal as shown in Fig. 1a. The results suggested that the reduction potential of alcohols became higher when the alkyl chain length increases. In other words, 1-octanol was identified as the best solvent for reducing Co and Cu metals (Fig. 1b). Experimentally, this reducing behavior of alcohols have been demonstrated in the synthesis of Cu based NSs. However, the formation of NWs requires milder reducing agents such as 1-heptanol to avoid the rapid growth of the nucleus into particles. On the other hand, alcohols with chain length shorter than 1-heptanol could not reduce Cu and Co.

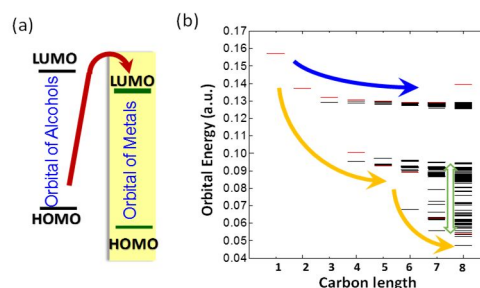


Fig.1 (a) scheme for orbital molecular calculation and reducing potential of alcohols.

**(b) Shape of metallic seeds:** The formation of multi-twinned particles (MTP) at earlier stages of reduction reaction facilitates the growth of seeds into NWs. Here, the oxidizing etching from Cl/O<sub>2</sub> pair on metallic seeds is vital for the generation of MTP and it is affected by parameters such as metallic chloride/acetate ratio, temperature, atmosphere, and surfactant.

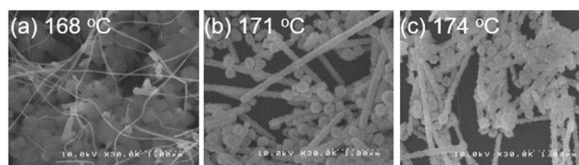


Fig.2 Effect of temperature in the synthesis of Cu-Co NSs.

Metallic acetates and chlorides are appropriated to synthesize Cu-Co NSs because acetate ions enhance the reducing ability of alcohols and chloride ions favor the etching mechanism. A molar ratio of 20:80 between Cu(II) chloride and Co(II) acetate is considered the best ratio to obtain a high yield of Cu-Co NWs. At different ratios, the yield decreased because the etching or reduction rate were higher than the optimized value.

For enhancing the yield of Cu-Co NWs, it is necessary to optimize the reaction temperature (Fig. 2). At 168 °C, the formation of spherical Co(OH)<sub>2</sub> particles was confirmed and Cu were reduced partially. At 171 °C, Cu-Co alloy NWs were obtained. Whereas at 174 °C, Cu-Co alloy NPs and NWs have been synthesized; at this temperature, the kinetics of the reaction is accelerated, and the formation of large particles are expected.

As mentioned above, an oxidant Cl/O<sub>2</sub> pair is formed from the metallic sources; however, due to Cu and Co are unstable in the air the reduction of the metallic sources should be carried out under N<sub>2</sub> atmosphere; consequently, the Cl/O<sub>2</sub> pair varied and affected the product as shown in Fig. 3. At N<sub>2</sub> flow rate lower than 100 mL/min, Cu and Co are not reduced, but Co ions form CoO NPs. At 100 mL/min, Cu and Co are not reduced, but here Co(OH)<sub>2</sub> NPs can be obtained. Up to 100 mL/min, Cu and Co NWs are reduced; here oxides or hydroxides are not observed.

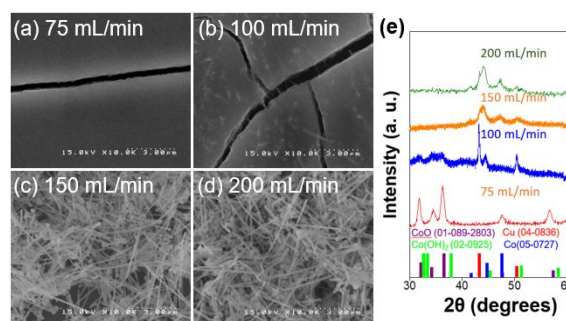


Fig.3 Effect of N<sub>2</sub> flow in the synthesis of Cu-Co NSs.

At the highest N<sub>2</sub> flow rate, the yield of NPs increased at the expense of the decrease in the yield of NWs.

**(c) The adsorption of surfactant:** The formation of Cu-Co NWs is favoured by the preferential adsorption of oleylamine on (100) planes through the N atoms. However, oleylamine could also be adsorbed on other planes if there is an excess.

Fig. 4 shows the products obtained at different amounts of oleylamine. At low amounts of oleylamine, although it can be adsorbed on Cu surfaces, the amount is not enough to avoid the particle growth in different directions and also to protect them from the oxidation and dissolution synthesizing Co(OH)<sub>2</sub> NPs and small quantities of Cu NWs. At higher amounts of oleylamine, the yield of NWs increases.

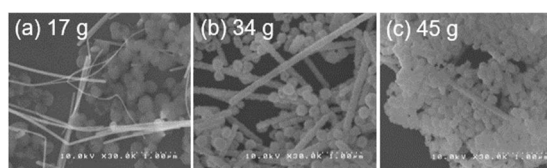


Fig. 4 Effect of the amount of oleylamine in the formation of Cu-Co NSs.

However, when there is an excess of oleylamine, > 41 g, the number of NPs increases due to the adsorption of oleylamine not only on (100) planes but also in the (111) planes. In contrast to low oleylamine amount, the particles are composed of metallic Cu-Co alloy. The FT-IR profiles show peaks of oleylamine at around 2850 and 2918 cm<sup>-1</sup> (symmetric and asymmetric vibration of alkyl chain), at 2952 cm<sup>-1</sup> (stretching vibration mode of CH<sub>3</sub>) and 3005 cm<sup>-1</sup> (C-H stretching mode).

Our results have shown that the synthesis of Cu-Co NSs could be controlled by parameters such as temperature, surfactant and atmosphere. However, here is not clear the reason for the formation of nanotubes (NTs) than NWs. Thus in the following section, the elucidation of the mechanism is attempted by monitoring the reaction.

**Mechanism formation of Co-Cu NWs:** The synthesis reaction was monitored by collecting samples at different temperatures and reaction times (Fig.5 ). Below 170 °C, the collected samples are unstable and dissolve in solution. However, due to the Co has similar behavior than Cu, the results obtained in Cu-Ni system can be extended to Co-Cu system (Ishijima 2018). At very low temperature, Co(II) and Cu(II)-amine complexes are formed. During heating Cu(II) is partially reduced to Cu(I) forming compounds as Cu(I)-amine complex whereas Co(II) amine complexes are transformed to layered hydroxides containing in their structure alcohol instead of OH molecules. At 170 °C, Co are reduced and, via galvanic reaction, Cu complexes are immediately reduced to Cu(0). The presence of Cl<sup>-</sup> ions and oleylamine favors the formation of multi-twinned Cu particles and subsequently their unidirectional growth. After that, Cu NWs are formed at 1h 50 min; here, fcc-Cu is the unique phase present. At 2h of reaction, the fcc-Cu remains as a unique phase, but the elemental mapping reveals that metal Co is deposited on Cu NW surface. At 2h and 10 min, a shoulder on (111) Cu peak corresponding to fcc-Co appears, while the elemental mapping shows that Co covers all the Cu NW surface. Up to 2h 10 min, the Co phases are predominant in the XRD curve, and the (111) Cu peak is shifted to a higher angle due to the alloying with Co. Whereas the elemental mapping shows that the Cu NW core is dissolved and diffuse in the Co shell forming a Cu-Co alloy NTs instead of NWs. In summary, the mechanism formation of Cu-Co NTs is proposed to follow as (Fig.6): (1) complexation of Cu and Co ions; (2) Cu reduction via galvanic reaction (3) Unidirectional growth of Cu (4) Deposition of Co on Cu NW and (5) Diffusion of Cu to Co shell.

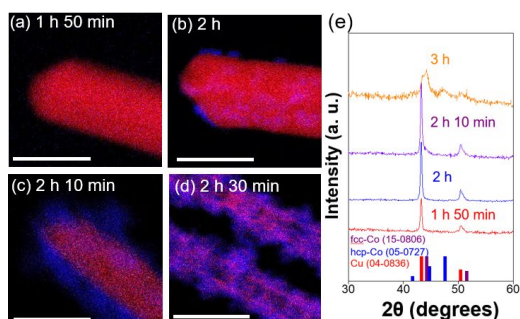


Fig. 5 (a-d) Elemental mappings and (e) XRD patterns of Cu-Co samples collected at different reaction time. Co (blue), Cu (red). Scale bar: 50 nm.

**Electrochemical Properties:** The transformation of CO<sub>2</sub> to formic acid using Cu base electrodes has been selected because the products could be applied in energy conversion, the reduction requires low energy, and the formation of byproducts, CO and H<sub>2</sub>, could be controlled. Electrochemical measurements were developed in the three-electrode electrochemical cell using NHCO<sub>3</sub> as an electrolyte . The voltammograms of Cu-Co NTs and Cu-Co NPs show peaks corresponding to the reduction of Co(II) at -0.95 V, CO<sub>2</sub> at -1.25 V, and H at -1.5 V. Here, the observed current density for Cu-Co NPs is higher than on Cu-Co NTs caused probably to their high specific surface. The chronoamperometric analyses reveal that the current density of Cu-Co NTs remained constant after 800 s, while it increased on Cu-Co NPs. Here, the reduction of Co(OH)<sub>2</sub> surface in Co increases the H<sub>2</sub> production, which is confirmed by chromatographic analysis. Fig. 7 shows the faradaic efficiencies for formate conversion. The optimum potential to reduce CO<sub>2</sub> into formate is -1.35 V for each catalyst where Cu-Co NTs has a higher faradaic efficiency than Cu-Co NPs. Under other potentials, the formation of hydrogen is preferred decreasing the faradaic efficiency. The difference of reactivity could be explained by the different surface states of the catalysts finding in the XPS analyses where Cu-Co NTs contain two times more CO<sub>3</sub>O<sub>4</sub> than Cu-Co NPs and Cu-Co NP surface is mainly constituted for Co(OH)<sub>2</sub>.

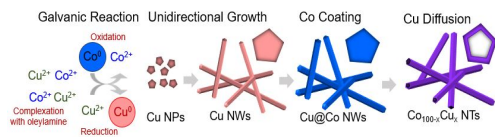


Fig. 6 Scheme of mechanism formation of Cu-Co NTs.

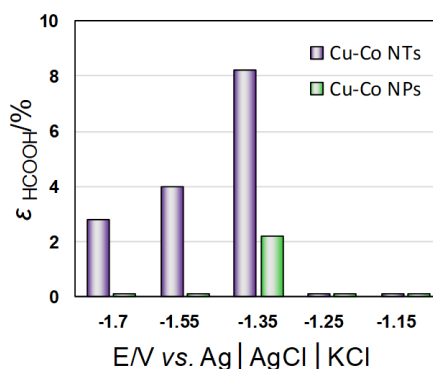


Fig. 7 Faradic efficiencies obtained during the electrochemical conversion of CO<sub>2</sub> to formic acid using Co-Cu NTs and NPs as electrodes.

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〔図書〕 計0件

〔産業財産権〕

〔その他〕

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6. 研究組織

	氏名 (ローマ字氏名) (研究者番号)	所属研究機関・部局・職 (機関番号)	備考
研究 分担 者	松本 高利  (Matsumoto Takatoshi)  (50343041)	東北大学・多元物質科学研究所・助教   (11301)	Contribution in theoretical calculation