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研究課題名（和文）Fine Structure of Bimetallic Solid Solution Nanoparticles Dispersed in Liquid by Double-Head Matrix Sputtering

研究課題名（英文）Fine Structure of Bimetallic Solid Solution Nanoparticles Dispersed in Liquid by Double-Head Matrix Sputtering

研究代表者
グエン　タンマイ（Nguyen, Thanh Mai）

北海道大学・工学研究院・助教

研究者番号：００７３０６４９

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研究成果の概要（和文）：この研究は、低揮発性液体または直接固体基板上にスパッタリングすることにより、バイメタル固溶体ナノ粒子の合成とそれらの微細構造の解析を目的とした。固溶体（Au / Ag）、金属間化合物（Au / Cu、Pt / Cu、Pd / Cu）または非混和性（Pt / Au）などのさまざまな金属ペアで、組成や大きさを制御できる固溶体合金ナノ粒子を得た。固溶体構造は、XPS、XRD、UV-Visの結果と組み合わせて、元素分布マッピングと組み合わせて確認された。サイズと合金組成の相関は、Pt / Auナノ粒子などで観察された。粒子の成長は、気液界面、液中、および保管中に観察された。

研究成果の学術的意義や社会的意義
この方法により、さまざまな酸化還元電位や粒子形成・成長の基本的な理解に留まらず、さまざまな金属ペアの固溶体合金ナノ粒子の制御可能で再現可能な合成が可能にする。そのため、従来の合成では困難だったマルチメタル固溶体合金の合成への応用が期待さレル。小粒径（<5 nm）で組成を調整可能な固溶体合金ナノ粒子は、粒子の特性を調整するための有用なツールであり、新しい特性を持つ触媒などの用途に期待される。

研究成果の概要（英文）：The research aims at the synthesis of bi-metallic solid solution nanoparticles by sputtering onto low-volatile liquid or directly on solid substrate and study of their fine structures. We obtained solid-solution alloy nanoparticles of controllable composition and sizes for various metal pairs such as solid-solution (Au/Ag), intermetallics (Au/Cu, Pt/Cu, Pd/Cu) or immiscible (Pt/Au) in the bulk. The solid-solution structure was confirmed with the atom resolution images coupled elemental distribution in combination with XPS, XRD, and UV-Vis results. The size-composition correlation was observed for alloy nanoparticles such as Pt/Au. Particle growth was evident on the gas-liquid interface, in the bulk liquid, and during storage.

研究分野：materials science and engineering

キーワード：solid-solution alloy　alloy nanoparticles　co-sputtering　polyethylene glycol　sputtering onto liquid　particle growth　immiscible　intermetallics

１．研究開始当初の背景

Metallic solid-solution alloy nanoparticles (NPs) and nanoclusters (NCs) are an important class of materials for applications in plasmonic, fluorescence, magnetics, and catalysts.[1] The advantages of alloy NPs emerge from the synergistic effect of different elemental components present in a single NP and the ability to modify and/or create their properties via varying their constituents' combination in terms of composition, structure, and size.[2,3,4]

Creating alloy NPs is challenging in a conventional chemical synthesis because the reduction order of metal salts is governed by their redox potentials.[5] In contrast, sputtering creates atoms/clusters via bombardment of the target by energetic gas ions. Thus, the method can produce particles of all metals with high purity.[6] Moreover, using a low vapor-pressure liquid as the substrate allows for the preparation of NPs in liquid dispersion.[7] There is plenty of room to modify the physicochemical properties of the liquid for varying NP characteristics for tuning their properties.[8-10] In particular, choosing suitable ligands we have successfully achieved tunable plasmonic/fluorescence Au, Ag, Cu NPs/NCs, strong visible absorption TiO NPs, and high index TiO_2 NPs in liquids.[10,11,12] The method can prepare alloy NPs in liquid regardless of their different reduction potentials. The alloy NPs were produced, e.g. Au/Ag, Au/Cu, but, sputtering has been limited within the use of a bulk alloy or bi/tri-elemental target.[9,10] Hence it is lack of versatility in tuning particle composition and fine-structures.

Recently, our double-target head for matrix sputtering allows for achieving Au/Ag alloy NPs in a large range of composition, thereby tunable plasmonic properties, via changing the applied current on each target.[13] This system offered a wider range of control of NP characteristics and properties via individual control of sputtering parameters for each target. Recent findings, that immiscible elements in the bulk state can mix to form solid-solution at the nanoscale,[14] serve as a basis to create alloy NPs. Using our sputtering system, we can study the formation of bimetallic NPs from different sources of metal atoms/clusters generated from different sets of metal targets such as metal pairs which form solid-solution, intermetallics or immiscible in the bulk. Monitoring the particle growth on solid and liquid substrates with varying the sputtering parameters can allow us to shed light on the particle formation and growth on the gas-liquid interface, the bulk liquid, and after sputtering. The obtained results of fine-structures of bimetallic NPs created from different metal pairs, their characteristics, formation, and growth mechanism are highly interesting and worth for study.

２．研究の目的

The research creates bimetallic solid-solution alloy NPs via the sputtering of double metal targets into a liquid medium. Based on investigation of different bimetallic systems, we aim to address the following questions: (**i**) from sputtered metal atoms/ clusters, is that possible to create bimetallic solid-solution NPs from metals which form intermetallic compounds (Au/Cu, Pt/Cu, Pd/Cu) and/or are immiscible (Au/Pt) in the bulk state; (**ii**) what is the fine structure of the bimetallic NPs and whether there is some limitation of size/composition for the alloy NPs to become segregated clusters or core-shell structures; (**iii**) can we control the experimental parameters to manage the particle growth and their formation and growth mechanism; (**iv**) Relation of NPs' size, composition, and assemble state of NPs in liquid with their structure and properties.

３．研究の方法

(1) Co-sputtering of Au/Cu NPs

A double-target head was used for sputtering. Liquid polyethylene glycol (PEG) with Mw = 600 was dried under vacuum heating then added to the petri dish located at the center of the sputtering chamber. The two metal targets were located symmetrically and headed to the liquid. During sputtering, the targets were cooled with a chilled solvent. The liquid PEG was stirred with a speed of 80 rpm. The sputtering time was set 30 min in Ar plasma under a pressure of 2 Pa. Co-sputtering onto C-coated Mo TEM grid for 30 s and sputtering of an Au/Cu alloy target (43 at % Cu) using only one head were conducted for comparison. The sputtering currents applied to Au and Cu target was varied from 0 to 50 mA.

(2) Co-sputtering of Pt/Au NPs

Similar sputtering experiments using the liquid PEG were conducted with varying the sputtering currents from 0-50 mA for Pt and Au targets. Besides sputtering onto the C-coated Cu TEM grid was conducted for various sputtering time (1-30 s).

(3) Sputtering of Pt/Cu NPs

Pt/Cu alloy target was used for sputtering using a single-head target. The sputtering parameters such as time, the rotation speed of the stirrer, temperature of PEG, and the sputtering period were varied to investigate their effect on the particle size and growth process.

Characterization: UV-Vis spectra were collected for the obtained NPs in PEG after sputtering. The transmission electron microscope (TEM) and scanning TEM (STEM) coupled energy-dispersive X-ray spectroscopy (EDX) were used to collect the images of the resulting NPs and the elemental distribution in single NPs. X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), X-ray florescence (XRF) and inductively coupled plasma-atomic emission spectroscopy (ICP-AES) were also used to analyze the NPs in terms of their crystal structure (XRD), electronic structures and chemical state (XPS), and compositions (XRD, ICP).

４．研究成果

(1) Solid-solution alloy NPs of tunable composition[15-17]

Au/Cu NPs have mean diameters of 1.7 to 2.5 nm when varying the sputtering currents of the target from 10-50 mA.[15] The composition increases linearly when increasing the sputtering current of one target and keeping the sputtering current of the other metal target constant. This indicates the capability to control the particle composition in a wide range. STEM-EDX and intensity line profiles of atom columns in high-angle annual dark-field (HAADF) images indicate that Au and Cu co-exist in the same NPs in each metal columns in a random fashion. There are not intermetallics or ordered structures can be observed. The results suggest the formation of solid solution Au/Cu alloy NPs. The relation of particle sizes of the alloy NPs and sputtering currents are not clear. Negligible aggregation in the Au/Cu alloy NPs was observed. Similar results were also obtained for co-sputtered Pd/Cu NPs.[16]

Au/Pt solid-solution NPs were also obtained by co-sputtering and their compositions were tunable by varying the sputtering currents applied to the two targets.[17] The XRD patterns of the obtained NPs clearly show peaks shift towards Au for NPs containing more Au. The lattice parameter obtained from XRD was linearly proportional to the composition of the constituent. The STEM-EDX mapping images show the distribution of Au and Pt in single NPs. Thus, the results indicate the formation of Au/Pt solid solution NPs.

It is worth mentioning that in this project solid solution alloy NPs were achieved by co-sputtering even though metal pairs form intermetallic compounds (Au/Cu and Pd/Cu)[15,16] or even immiscible (Au/Pt)[17]

in the bulk. This indicates that the method is very powerful for making solid-solution NPs of tunable composition and sizes.[18]

(2) The composition-size relation in solid-solution alloy NPs[15-17]

The following results were obtained under the sputtering time of 30 min and sputtering currents varied from 10 to 50 mA onto PEG. The relation of the alloy compositions and particle sizes was not very obvious in the sputtered alloy NPs such as Au/Cu and Pd/Cu.[15,16] However, it is found that Pt/Au alloy NPs became smaller for higher Pt content and bigger for lower Pt content. An additional interesting finding is that the alloy NPs have similar sizes for the same Pt content regardless of the sputtering currents. The composition, i.e., Pt content also impacts the agglomeration state of the samples wherein large agglomerations formed when Pt less than 16 at % and separated NPs and stable colloidal dispersions, i.e., for months, were obtained when Pt more than 26 at %. The stronger interaction of PEG to Pt and larger formation energy of Pt can explain the phenomenon. The experimental results also suggest possible selective attachment mediated growth can occur in PEG. This causes the final alloy NPs with somehow higher Pt on the surface to be stabilized and to stop growing. Thus, particles of the same nominal Pt content can grow to the same sizes.

(3) NP growth mechanism[19]

It is found that the temperature of liquid PEG in range or 20 to 50 °C had a negligible impact of the particle sizes of Cu/Pt alloy NPs (Pt of 29 at%). The particle size increased obviously by prolonging the sputtering time, e.g., from 0.5 h to 4 h with the formation of polycrystal particles via secondary particle collision and growth, suggesting the particle growth in the bulk PEG liquid. The particle growths on the PEG surface to some extent were also evidenced. This is judged from the fact that for the same total sputtering time the shorter sputtering period (for particles falling into the bulk PEG) resulted in smaller particle size. Besides, for sputtering onto PEG, a higher stirring speed was found to help decrease the particle size. It is found in another study that a certain growth of particle further occurred when the dispersions were stored after sputtering via consuming of free metal atoms/clusters in PEG.[20] The results in this study contribute to the basic understanding of the formation and growth of NPs produced via sputtering onto liquid PEG, allowing for managing sputtering parameters to control the particle size.

(4) Oxidation stability of the alloy NPs and the role of PEG

For the sputtered alloy NPs of noble metals such as Au/Pt, the NPs were stable from oxidation.[17] However, the surface oxidation was detected to a certain extent when Cu is one component of the binary solid solution alloy NPs such as Pt/Cu, Au/Cu, and Pd/Cu.[15,16,18,19]

It is also found that PEG has an important role in making NPs of small sizes (typically below 5 nm for our sputtering conditions). The particles obtained by sputtering onto TEM grids had particle sizes increase rapidly with time.[17,19,20] Within tens of second to several minutes sputtering on the grids, the particle sizes were over that of NPs obtained in PEG for 30 min sputtering. This emphasizes the advantage of combining vacuum sputtering and a liquid substrate in producing NPs. The interaction of PEG to different metal and alloys can be different, resulting in the different colloidal stability of the NP dispersion.

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５．主な発表論文等

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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