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The effect of 3D silver nanodome size on hydrogen evolution activity in alkaline solution[☆]

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ABSTRACT

Three-dimensional (3D) Ag nanodomains (AgNDs) having different sizes (400, 800, 1200 and 1600 nm) were fabricated using combination of nanosphere lithography and soft lithography. The surface structures of 3D assembled latex particles, nanovoids and metal nanodomains (ND) were examined using scanning electron microscopy (SEM). Their heights and widths analyses were performed with the help of atomic force microscopy (AFM). The effect of diameter of the NDs on their hydrogen evolution activity was examined in 6 M KOH solution at 298 K using electrochemical techniques. Their activities were compared with the activity of bulk Ag electrode. The preparation of 3D-AgNDs having various diameters and examination of their size effects on the water splitting activity have not been studied yet and are being reported firstly. It was found that very well-structured and very uniformly distributed NDs can be fabricated using this procedure. AgNDs exhibit higher hydrogen evolution activity with respect to bulk Ag. Their hydrogen evolution activity depends on their diameters; 1200 nm NDs were the best among them. The current density at -1.40 V(Ag/AgCl) which is proportional to the rate of hydrogen releasing reaction increases from 0.70 mA cm⁻² to 44.13 mA cm⁻² at this ND electrode with respect to the bulk Ag electrode. At the same 3D-AgNDs electrode and potential, the resistance against the HER reduces from 148.7 Ω cm² to 1.12 Ω cm² (99.6%) by comparing with the bulk Ag electrode. The average surface roughness factors of bulk Ag, 400 nm, 800 nm, 1200 nm and 1600 nm AgNDs are 8, 123, 100, 291 and 176, respectively. The superior hydrogen evolution performance of this electrode is related to its well-structured surface and large real surface area.

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Introduction

Hydrogen is a fully recyclable green energy carrier with a practically unlimited supply [1–4]. Although there are many

production methods, electrolysis of water is one of the best and simplest alternative clean and renewable hydrogen production way for this aim since most of production techniques are not green. Therefore, studying hydrogen evolution reaction (HER) has become very popular [5]. Electricity generated

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from renewable energy sources such as nuclear, solar or others can be applied to split water and produce hydrogen gas with high purity and large-scale. Unfortunately, this method is not ready for large-scale commercial applications due to high energy consumption in electrolysis system which arises from overpotentials at electrodes and electrolyte. In order to overcome these drawbacks it is needed to reduce cost production of hydrogen by lowering anodic and especially cathodic overpotentials as well as choosing cheap electrode materials [6].

Pt is the best electrocatalyst for the HER. However, its practical application for this aim is not common currently due to its low abundance on the earth and high cost [7,8]. In literature [9–22], there are many approaches to lowering amount of Pt or Pt-group metals for fabrication of electrodes by modifying different substrates. However, further and satisfying studies are needed. Therefore, efforts are being devoted to develop cheaper and efficient earth-abundant metals alternative to noble metal-based electrode materials [23,24]. By comparing with the other platinum-group metals, Ag has lower cost and higher abundance on the earth. The literature survey has indicated that Ag nanostructures as pure or its alloys have promising electrochemical activity for cleaving of water to hydrogen and oxygen gases [11,25–27]. The enhanced hydrogen evolution activity of nanostructures was assigned to their larger real surface area [28]. In addition to type of metals used for fabrication of electrodes, enhancing real surface area of electrocatalyst is one of another efficient ways of increasing hydrogen evolution efficiency.

The use of nanomaterials *i.e.* nanoparticles, nanowires, nanotubes or nanoflowers provides a larger active surface area [6,29]. AgNPs have been prepared using various techniques [11,28,30,31]. Recently, we have suggested a new approach to fabricate reproducible, size-controllable and simple method to fabricate nanodome structures [32,33]. The method contains combination of soft lithography, nanosphere lithography and electrochemical deposition. To our best knowledge, the preparation of 3D NDs having various diameters and examination of their size effects on the water splitting activity have not been studied yet and are being reported firstly.

In this study, we have fabricated 3D-AgNDs having various diameters by the combined method and studied the effect of ND sizes on the HER since this reaction is a surface process and closely depends on real surface area as well as size of particles. The hydrogen releasing activity of the nanostructures was evaluated in 6 M KOH solution. For this aim, polarization and EIS techniques were used. The surface structures of latex particles assembled on glass slides, nanovoids fabricated on PDMS template and 3D NDs were examined by SEM and AFM techniques. We have aimed to fabricate a new cheap, electrochemically active and size-controllable nano surfaces to be used for water splitting. It is expected that the procedure applied here contribute to literature since nanosurfaces, which have superior HER activity than bulk metal, is fabricated by this method and such surfaces can also be used as supporting materials for further modifications by loading low dosage of platinum group metals.

Materials and methods

This is a second and improved part of our published results [32]. In the first part of this continued study [32], we applied a new, cheaper and simpler method without adhesion layer, which includes combination of soft lithography and nanosphere lithography as well as electrochemical deposition, to fabricated 3D-AgNDs. The preparation procedures were given in detail in the former studies [32,33]. Therefore, the fabrication procedures will not be repeated in this study again. In this study, 3D-AgNDs having various diameters (400, 800, 1200 and 1600 nm) were fabricated by applying the same procedure to determine the best ND size for alkaline water electrolysis since the diameter of nanoparticles is one of the main factor affecting electrocatalysis. To change the diameter of NDs, latex particles having different diameters were used. In summary; (i) Spherical latex particles having 1600, 1200, 800 and 400 nm diameters were packed on a glass slide separately by a method described in literature [34,35]. The latex spheres concentration, stage velocity and the latex particles volume dropped between to glass slides were 0.8% , $1 \mu\text{m s}^{-1}$ and $40 \mu\text{L}$, respectively (ii) Polydimethylsiloxane (PDMS) polymer mixture was poured on the top of the latex thin films and cured at 70°C for 1 h. The cured PDMS was peeled off, then washed with dichloromethane sufficiently and nanovoids having different diameters were obtained on the PDMS surfaces. (iii) The nanovoids were filled with Ag and 3D-AgNDs having various sizes were fabricated. Before metal deposition, surface of PDMS was deposited with a very thin Cr layer by physical vapor deposition (PVD) technique to make it conductive and enhance adherence of metal to the substrate. The Ag deposits were galvanostatically prepared in $0.1 \text{ M AgNO}_3 + 0.1 \text{ M KNO}_3 + 1 \text{ M NH}_3$ solution by applying a constant 3.5 mA current to the electrolysis system until ensuring all of the nanovoids were filled with Ag ($\sim 4 \mu\text{m}$). (iv) The backside of NDs was thickened by electrodeposition of a thicker and harder Ni layer to enhance its mechanical strength. Nickel electrodeposition was performed galvanostatically by applying 2.5 mA for 1800 s and followed 10 mA for 5400 s. The chemical composition of deposition bath was $30\% \text{ NiSO}_4 \cdot 7\text{H}_2\text{O}$, $1\% \text{ NiCl} \cdot 6\text{H}_2\text{O}$ and $1.25\% \text{ H}_3\text{BO}_3$. (v) The thin AgNDs sheets were cut using a lancet in dimensions of $2 \text{ mm} \times 10 \text{ mm}$. The tip of the samples was attached to a stainless steel forceps, coated with poly ester except $2 \text{ mm} \times 2 \text{ mm}$ surface area that contains NDs and working electrodes were fabricated. As it was stated above, the details can be found in the published literature [32,33].

For comparison and show superiority of NDs, a bulk Ag electrode having exposed surface area of 0.28 cm^2 was used. The bulk Ag electrode was cut from a cylindrical Ag rod to a length of about 5 cm and coated with polyester block except only its bottom surface, which was exposed to the electrolytes. The electrical conductivity was provided by a copper wire [32]. The surface of Ag electrode was polished with emery papers having different grade sizes which ended with 1200 grid. Afterwards, the electrodes were treated with distilled water, washed with absolute ethanol, dried under N_2 gas and immersed into the test solution immediately. Working electrodes were electrochemically activated before measurements.

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