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Bismuth nano-powder electrode for trace analysis of heavy metals using anodic stripping voltammetry

Gyoung-Ja Lee, Hi-Min Lee, Chang-Kyu Rhee *

Nuclear Nanomaterials Development Laboratory, Korea Atomic Energy Research Institute (KAERI), 150-1 Deukjin-dong, Yuseong-gu, Daejeon 305-303, Republic of Korea

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Abstract

In the present work, a more sensitive and conveniently usable electrode sensor for a trace analysis of heavy metal was developed by using Bi nanopowder synthesized by levitational gas condensation (LGC) method. It was observed from the TEM image that the Bi nanopowder is spherical in shape with a size of nearly 50 nm. The XRD pattern revealed intense peaks which can be indexed as a rhombohedral structure of Bi without any other diffraction peaks corresponding to an oxide or an impurity. This indicates that the resulting nanopowder synthesized by the LGC method is a highly crystallized Bi with a high purity. The square wave anodic stripping voltammograms (SWASV), experimentally measured for the Bi nanopowder electrode, showed well-defined and highly reproducible electrochemical responses relating to the stripping of Cd and Pb. The detection limit of the electrode was estimated to be 0.15 μg/l and 0.07 μg/l for Cd and Zn, respectively, on the basis of the signal-to-noise characteristics ($S/N = 3$) of the response for the 1.0 μg/l solution under a 10 min accumulation.

Keywords: Bi nanopowder electrode; Levitational gas condensation method; Heavy metal; Square wave anodic stripping voltammetry

1. Introduction

Recently, a Bi electrode for a trace analysis of heavy metal has attracted considerable attention as a new alternative to the common mercury electrode due to its low-toxicity, excellent resolution of neighboring peaks (e.g., Cd, Pb, Zn, Tl, etc.) and insensitivity to the dissolved oxygen in a solution [1–11]. Bi film-coated carbon electrode has generally been used as an electrode. However, for a wider application of the Bi sensor, there are still some problems to solve: comparably low detection limit and an additional washing or polishing step of the carbon surface. Furthermore, there are instances where the preparation of pre-plated Bi film electrode or the introduction of Bi ions into a solution is undesirable or impossible.

In order to overcome the above weaknesses of the Bi film electrode, it is necessary to develop a Bi nanopowder electrode with a larger electrochemical active surface area, which is directly applicable to a trace metal analysis without a pre-deposition step of Bi in a Bi-containing solution. The levitational gas condensation (LGC) method is one of the most promising methods for the synthesis of various metal or oxide nanopowders [12–15]. While the other methods such as a magnetron sputtering, melt spinning, and mechanical alloying have more complex procedures, the LGC method is a simple one-step process to fabricate nanopowders with a more uniform particle size distribution. In our previous work, a complicated levitation and evaporation mechanism for a nanopowder fabrication was explained in detail [13–15].

Under these circumstances, in this work, in order to develop a flexible and conveniently usable Bi sensor with a higher sensitivity, Bi nanopowder was prepared by the LGC method, and then coated onto the conductive carbon

* Corresponding author. Tel.: +82 42 868 8551; fax: +82 42 868 4847.
E-mail address: ckrhee@kaeri.re.kr (C.-K. Rhee).

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layer which was pasted onto the flexible polymer film by the screen printing method. The square wave anodic stripping voltammograms (SWASV) were experimentally measured for the Bi nanopowder electrodes as a function of the Pb and Cd concentrations. Then, their detection sensitivities and detection limits were quantitatively estimated.

2. Experimental

2.1. Preparation of a flexible carbon/Bi nanopowder electrode

Bi nanopowders were synthesized by the LGC method using a micron powder feeding (MPF) system. The apparatus consisted of a high frequency induction generator of 2 kW, a levitation and evaporation chamber and an oxygen concentration control unit. The amount of micron powder fed into the system was controlled at 20 mg/min. The Ar gas flowed into the chamber with a rate of 1.4 l/min in order to maintain the pressure in the chamber at 70 torr. From the transmission electron microscopy (TEM) image in Fig. 1a, it was observed that the Bi nanopowder is spherical in shape with a size of nearly 50 nm.

In order to prepare the working electrode for the electrochemical measurement, conductive carbon ink (Dong-Young Chemical Co., Ltd., Korea) was pasted with a thickness of 80 μm onto a flexible polyester film using the screen printing method, and then partially covered by an insulating layer, as illustrated in Fig. 1b. Bi nanopowders of 0.0025 g were dispersed well into 50 ml distilled water using an ultrasonic vibrator. For a strong adhesion of the Bi nanopowder onto the carbon paste, 10% Nafion solution was added into the suspension. Finally, 10 μl of the Bi-containing suspension was dropped onto the working area and dried in air at 298 K over 12 h. From the SEM image of the working area in Fig. 2, it was found that the Bi powders were homogeneously distributed onto the electrode surface.

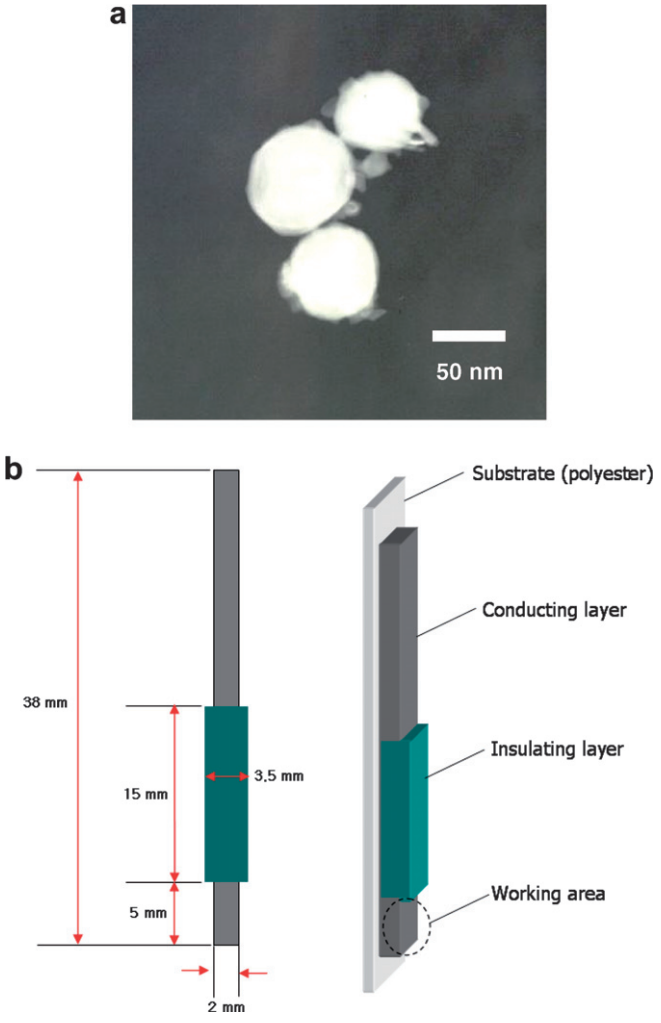

Fig. 1. (a) Transmission electron microscopy (TEM) image of the bismuth nanopowder synthesized by the LGC method and (b) schematic diagram of the working electrode.

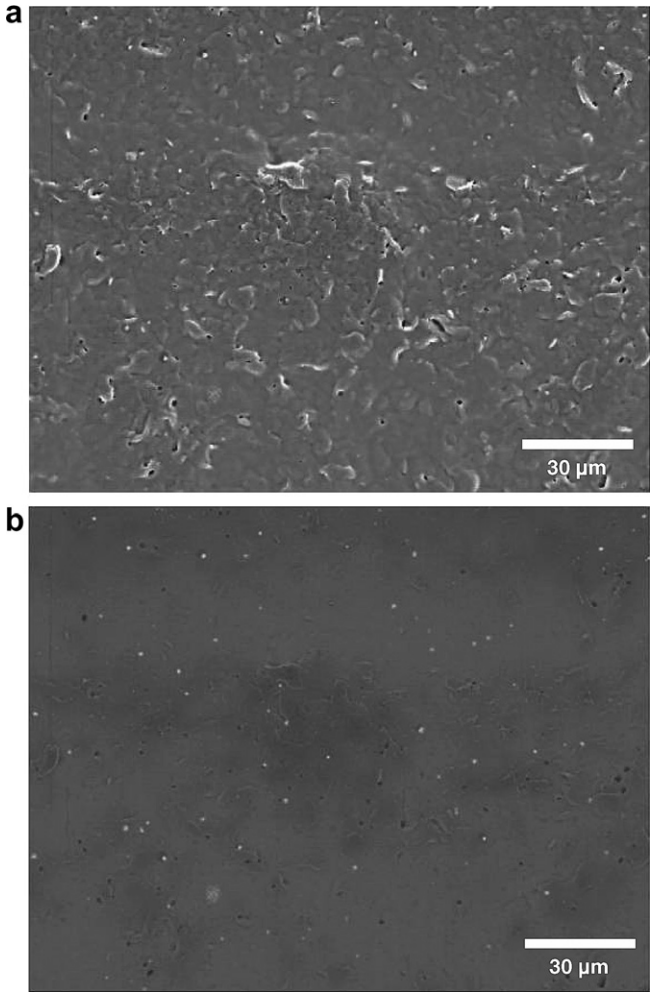

Fig. 2. (a) Scanning electron microscopy (SEM) image and (b) backscattering image of the electrode surface.