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Site-selective chemical etching of GaAs through a combination of self-organized spheres and silver particles as etching catalyst

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Abstract

Microfabrication of the *n*-GaAs substrate surface was investigated by a combination of colloidal crystal templating, electroless plating and subsequent metal-assisted etching using noble metals as a catalyst. Ag and Cu nanosized particles were deposited site selectively to form metal-honeycomb patterns on GaAs using self-organized polystyrene spheres as a mask. By Ag-assisted etching, GaAs was effectively etched into a convex-array structure. Different anisotropic etching patterns were observed throughout the substrate after Ag-assisted etching, by changing the crystal-face orientation of *n*-GaAs from (1 0 0) to (1 1 1).

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1. Introduction

To fabricate multifunctional porous Si, “metal-assisted chemical etching” was proposed as a useful technique [1]. The method utilizes the catalytic ability of noble-metal thin films/particles in an etchant composed of a mixture of HF and H_2O_2 solutions without the use of any electrical connections or strong oxidizing agents [2]. Thus, the effects of metal-assisted etching with Au, Pt, Ag and Pd were systematically investigated for Si (1 0 0) [2,3]. However, the investigation of this etching method in group III–V compound semiconductors is rare in contrast to the case of Si, so it is less understood. Therefore, we describe here the effectiveness of metal-assisted etching for the most widely utilized group III–V compound semiconductor, GaAs, as a function of crystal-face orientations, i.e., (1 0 0) and (1 1 1) possessing a resistivity level of $10^{-3}\ \Omega$ cm order. The morphological control of the GaAs surface is of interest from the standpoint of fundamental science, and further, the application to devices in the future.

A templating technique based on self-organized polystyrene (PS) spheres has been reported for the fabrication of Si hole arrays [4], dot arrays [4], disk arrays [5], pillar arrays [5] and porous alumina possessing 2D/3D composite structures [6] by means of anodization as well as the control of pit initiation sites on aluminum [7]. Here, PS spheres can be dissolved easily by ultrasonic cleaning in toluene after the experimental processes. Therefore, PS spheres forming the self-organized structure were selected as a mask, aiming for the “site-selective” etching of GaAs. As a result of previous studies [8,9], electroless Cu plating on Si has been clarified and the technique was well established. Also, deep cylindrical nanoholes have been generated by etching the *p*-Si substrate by depositing Ag particles [10], however, there is no report on successful electroless Cu and Ag plating of GaAs. Thus, Cu and Ag were chosen as metal sources for the metal-assisted etching in the present study.

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2. Experimental

A GaAs wafer was cleaved to 1 cm × 1 cm in size and used as a substrate. Before forming a mask on the substrate, the native-oxide film was removed by immersing the substrate in 46 wt% HF solution for 15 min. Schematic explanation of the present experimental procedure and site-selective chemical etching of GaAs is shown in Fig. 1. A solution with PS spheres of 1 μm diameter was adjusted to the suspension concentration of 0.25%, supplied drop-wise onto the substrate, and then dried in air. After the complete evaporation of the solvent, PS spheres forming a close-packed structure were obtained as a mask. To fix the mask on the substrate, a heat treatment was carried out at 100 °C for 1–2 h (Fig. 1a). Subsequently, electroless Cu plating was proceeded in a solution consisting of 0.01 wt% $CuSO_4 \cdot 5H_2O$ and 46 wt% HF without any reductants [8,9], with the bath temperature maintained at 25 °C for 12 ∼ 20 min. Immersion plating in an aqueous solution containing 10^{-4} mol dm^{-3} $AgClO_4$ and 10^{-3} mol dm^{-3} NaOH at room temperature was conducted for 20 min [10] for Ag deposition (Fig. 1b). It was immediately followed by chemical etching in a mixture of 5 mol dm^{-3} HF and 1 mol dm^{-3} H_2O_2 solutions at room temperature (Fig. 1c–e). Finally, ultrasonic cleaning in toluene was carried out to remove the mask. The obtained surface morphologies of GaAs were observed by scanning electron microscopy (SEM, Hitachi S-4200) and atomic force microscopy (AFM, Digital Instrument Nano Scope IIIa).

3. Results and discussion

Fig. 2 represents a SEM image of a Cu-honeycomb pattern formed on the *n*-GaAs (1 0 0) substrate by electroless plating for 16 min. Cu particles were deposited site selectively only on the exposed areas of the substrate, i.e., the apertures between PS spheres, whereas essentially no Cu particles were deposited at the contact areas of the PS sphere and the underlying GaAs substrate. It was found that the Cu-honeycomb pattern consists of an aggregation of Cu particles of ∼75 nm on average, though the size of particles is somewhat scattered. The present study could be the first report on Cu plating of GaAs without bias because the surface processing technique of GaAs has not

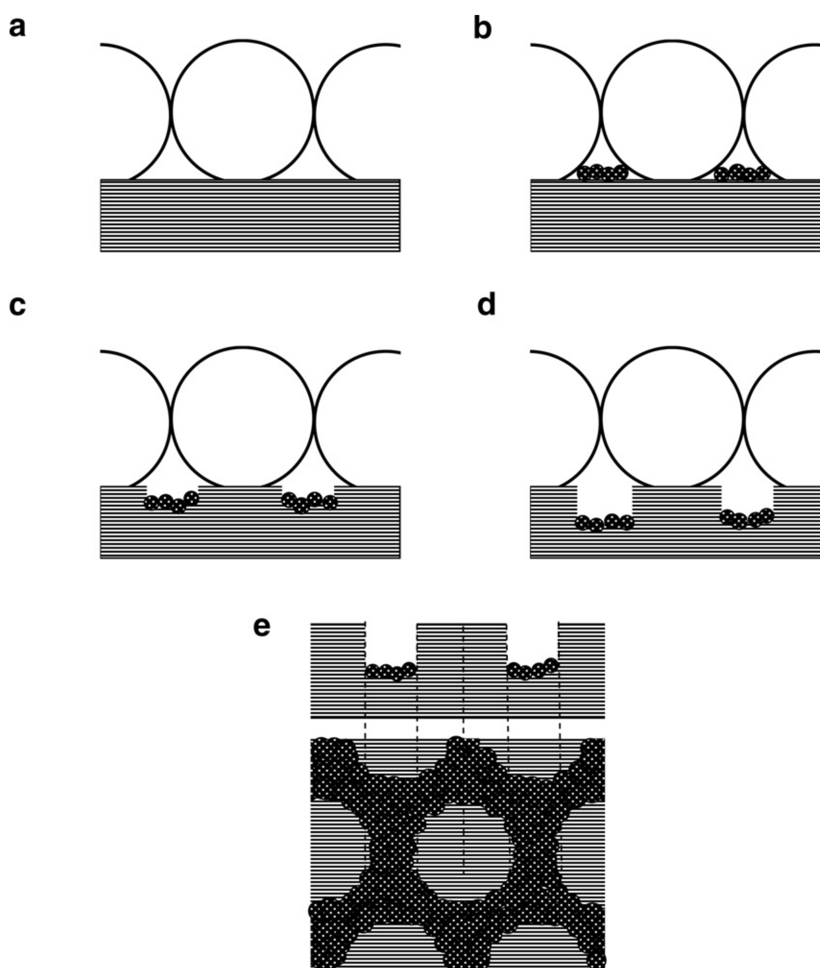

Fig. 1. Schematic explanation of site-selective chemical etching of GaAs. (a) Colloidal crystals formed on substrate as a mask, (b) deposition of noble-metal particles, (c) the initial stage of chemical etching of GaAs using a metal catalyst, (d) after completion of chemical etching, and (e) obtained cross section and corresponding top view after removal of the mask.

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