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Fabrication of Size Controlled Nanohole Array on III-V Semiconductor substrate by ICP-RIE using Nanoporous Alumina Mask

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Abstract. Formation of size controlled nanohole arrays on semiconductor substrate can be used variously in applications of photonic and electronic device. The unique structure of nanoporous alumina was directly used as an etching mask for pattern transfer into the GaAs substrate. Using the alumina masks prepared at the anodic voltage of 24 V in 0.3 M sulfuric acid solution and 40 V in 0.3 M oxalic acid solution, fabricated were the arrays of nanohole on GaAs substrate by inductively coupled plasma reactive ion etching (ICP-RIE). The etching was conducted in a $SiCl_4$/Ar mixed gas system. The uniform nanohole arrays were formed as replica of ordered lattice pattern of the mask. Depending on property of the alumina mask used, the size of nanohole was controlled to have hole diameter of 60 nm (with the hole density of 1.0×10^{10} cm^{-2}) and of 30 nm (with the hole density of 2.7×10^{10} cm^{-2}), respectively. So, the alumina mask attached on the GaAs substrate mainly determines the distribution and the size of the nanohole arrays. ICP-RIE using nanoporous alumina masks can control the size and the density of nanohole on compound semiconductor substrate.

Introduction

Nanoporous alumina layer has been very attractive because of their potential applications for nanometer devices. The geometry of nanoporous alumina is schematically represented as a close-packed array of columnar hexagonal cells, each containing a central pore normal to the substrate [1]. Porous alumina with long and narrow holes can be produced, the diameter of which is variable between 10 and 95nm by changing the preparation procedure [2]. The nanoporous alumina has been used as a template in order to grow nanomaterials such as nanodots [3] and carbon nanotubes [4]. The growth of highly ordered quantum dot arrays of InGaAs on the GaAs substrate has been successfully achieved using nanoporous alumina as evaporation mask [5]. Also, the structure of nanoporous alumina has examined as a transfer mask for pattern of nanometer size into the semiconductor substrates [6-9]. Reactive ion etching (RIE) has been extensively used for manufacturing of micrometer devices. This RIE patterning method using nanoporous alumina mask provides efficient and cost effective alternative approaches for realizing ordered nanostructures [6].

In this work, we report the fabrication of size controlled nanohole arrays on GaAs substrate by inductively coupled plasma reactive ion etching (ICP-RIE) using nanoporous alumina as etching mask. The etching was conducted in a $SiCl_4$/Ar mixed gas system. The ion bombardment induced by ICP-RIE responses effectively on GaAs substrate through the nanoholes of the alumina.

Experimental Procedure

An aluminum foils (99.99%, 100μm thickness) were conducted electropolishing in a mixed solution of perchloric acid (60%) and ethanol (1:4 in volume) at constant voltage condition of 20 V at

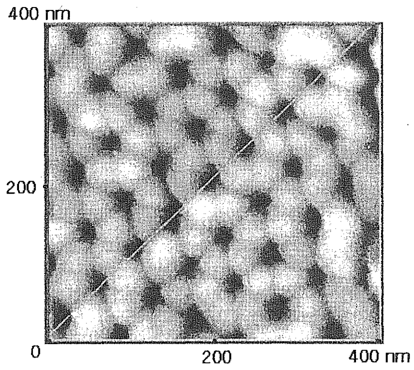
3 °C for 60 sec. During anodization, the electrolyte was vigorously stirred and maintained at 3 ± 0.1°C using a jacket beaker in a iso-temperature circulator system. The first anodization was performed by applying the DC voltage of 40 V in 0.3 M oxalic acid solution and 24 V in 0.3 M sulfuric acid solution, respectivlely. The alumina layer formed by first anodization was completely dissolved away in a mixture solution of phosphoric acid (0.4 M) and chromic acid (0.2 M) at 65 °C for several hours. Then, a second anodization was conducted under the same condition as the first anodization except the anodization duration. Well-ordered porous alumina layer was grown on the hexagonal texture formed from the first anodization. After the second anodization, the remaining aluminum substrate was removed in a saturated $HgCl_2$ solution. After the lift-off process, the alumina barrier layer at the bottom of the cylindrical channels was uniformly etched out in an aqueous 5 wt% phosphoric acid at 30°C, sequentially.

The alumina mask with through-hole was put on a semi-insulating GaAs (100) substrate. This alumina mask/GaAs substrate was subsequently placed on the cathode electrode of ICP-RIE system. Under the fixed flow rate of the etching gases $SiCl_4$: Ar (15: 5), GaAs etching was conducted under ICP source power of 500 W and rf bias power of 60 W. This etching condition resulted in GaAs etching rate of 0.59 μm/min. After ICP-RIE, the alumina mask bonded on GaAs substrate was dissolved out in mixture of phosphoric acid and chromic acid for several hours at 65 °C.

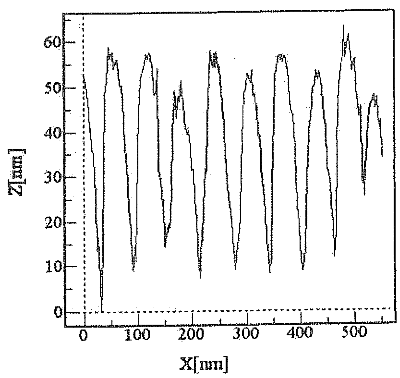
The morphology of nanoporous alumina mask and GaAs surface was observed using a field emission scanning electron microscope and an atomic force microscope (AFM).

Results and Discussion

We obtained well-ordered nanoporous alumina from aluminum foil by two-step anodization process[10]. The close-packed hexagonal cell size and pore diameter of alumina can be controlled by anodic voltage, type of electrolyte and temperature [2,3]. At low anodization voltages in a sulfuric acid electrolyte, a nanoporous alumina masks with a high pore density can be prepared. Plane-view and section analysis of AFM image of the alumina mask prepared at an anodization voltage of 24 V in a sulfuric acid solution are shown in Fig. 1. The hexagonal cell size and an average pore density of the alumina mask was 65 ± 5 nm and 2.7 (±0.2) × 10^{10} cm^{-2}, respectively. The pore size of the alumina mask was adjusted in range of the hexagonal cell size by chemical etching time. The section analysis of the AFM image of the alumina mask in Fig. 1(b) shows well-defined periodic nanohole profile.

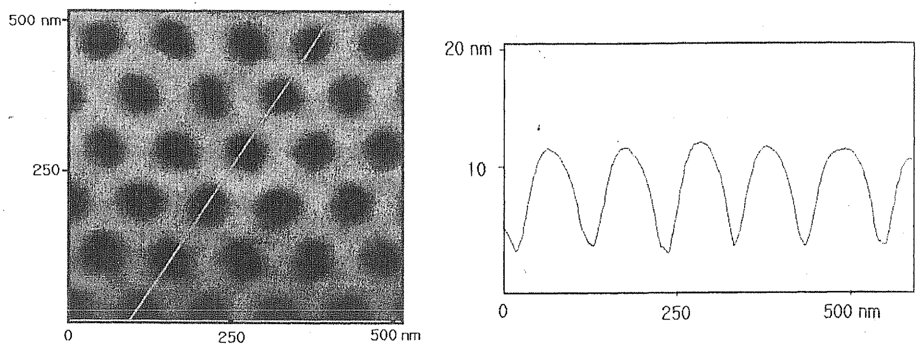


(a)



(b)

Fig.1. (a)Plane-view and (b) section analysis of AFM image of the alumina mask prepared at 24 V in 0.3 M sulfuric acid solution.



(a) (b)

Fig. 2. (a) Plane-view and (b) section analysis of AFM image of the nanoholes formed on GaAs substrate by ICP-RIE using the alumina mask prepared at 40 V in 0.3 M oxalic acid solution.

We investigated nanohole fabrication process on GaAs substrate by inductively coupled plasma reactive ion etching (ICP-RIE) using the nanoporus alumina masks prepared at different anodization conditions. The etching was conducted in a $SiCl_4$/Ar mixed gas system. The GaAs substrate kept etching for 15 sec by ICP-RIE using the nanoporous alumina mask. The alumina mask bonded on GaAs substrate was dissolved out in chemical etching solution. After removal of the alumina mask, AFM image of the GaAs surface morphology are shown in Fig. 2. The ion bombardment induced by ICP-RIE responses effectively on GaAs substrate through the nanoholes of the alumina. The section analysis of AFM image in Fig. 2(b) clearly shows the uniform nanoholes with the average peak depth of ca. 9.0 nm.

Depending on property of the alumina mask used, the uniform array of nanohole was formed as replica of ordered lattice pattern of the alumina mask. Plane-view SEM image of the nanohole array formed for 150 sec by ICP-RIE using the alumina mask that was performed by applying a constant DC voltage of 24 V in 0.3 M sulfuric acid is shown in Fig. 3. SEM image of the surface morphology of the etched GaAs substrate in Fig. 3 clearly shows that the average diameter and the density of the nanohole formed on GaAs substrate are 30 ±5 nm (with hole spacing of 65 nm) and 2.7×10^{10} cm^{-2}, respectively. Plane-view SEM image of the nanohole array formed for 150 sec by ICP-RIE using the alumina mask that was performed by applying a constant DC voltage of 40 V in 0.3 M oxalic acid is shown in Fig. 4. The average diameter of the nanohole formed on GaAs substrate in Fig. 4 is 65 ±5 nm (with hole spacing of 105 nm) and the nanohole density is 1.0×10^{10} cm^{-2}.

Well-ordered nanoporous alumina masks play a very important role in determining formation of nanohole array on the GaAs substrate. So, the alumina mask that controlled by anodization conditions determines the diameter and the density of the nanohole. This indicates that reduction in the size and increasing the density of compound semiconductor nanohole array can be possible by using nanoporous alumina mask. The shape of the etched GaAs nanohole depends not only on the shape of the mask but also on the conditions of ICP-RIE, such as species of etching gas, etching time, and ICP source power / rf bias power. This patterning method may provide alternative approach for fabrication of nanostructures that can be used variously for nanotechnology.

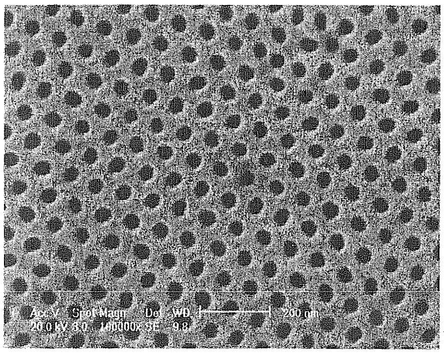


Fig. 3. Plane-view SEM image of the GaAs nanohole array was formed by ICP-RIE using the alumina mask that was performed by applying a constant DC voltage of 24 V in 0.3 M sulfuric acid.

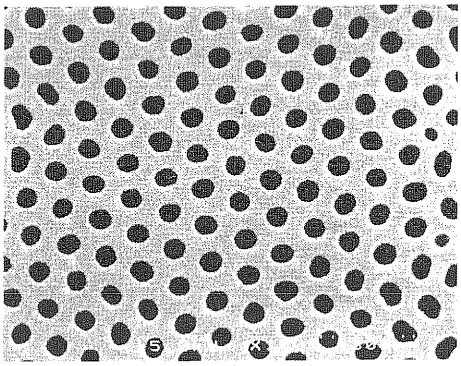


Fig. 4. Plane-view SEM image of the GaAs nanohole array was formed by ICP-RIE using the alumina mask that was performed by applying a constant DC voltage of 40 V in 0.3 M oxalic acid.

Summary

A uniform array of nano-sized pores produced in porous alumina was transferred on GaAs substrates by inductively coupled plasma reactive ion etching (ICP-RIE). The unique structure of nanoporous alumina was directly used as a mask for pattern transfer into the GaAs substrate. Using an alumina mask, the uniform GaAs nanohole arrays were formed as replica of ordered lattice pattern of the mask. Depending on property of the alumina mask used, the size of nanohole was controlled to have hole diameter of 60 nm (with the hole density of 1.0×10^{10} cm^{-2}) and of 30 nm (with the hole density of 2.7×10^{10} cm^{-2}), respectively. The alumina mask controlled by anodization conditions such as anodic voltage and type of electrolyte, mainly determines the distribution and the size of the nanohole arrays. ICP-RIE using nanoporous alumina masks can control the size and the density of nanohole on compound semiconductor substrate.

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