Articles

Fabrication of Microstructures by Wet Etching of Anodic Aluminum Oxide Substrates

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Anodic aluminum oxide (AAO) was used as substrates of microstructure fabrication. Complex structures with high aspect ratios were obtained by the single-step wet etching of the AAO substrates. Structures with different shapes and aspect ratios could be simultaneously obtained with vertical sidewalls. The surface morphology of the fabricated structures could be tailored. It is expected that various MEMS/NEMS devices can be built using this technique.

Introduction

Microfabrication is the basis of the information revolution in the 20th century. It is also expected to be the enabling technology for the NEMS/MEMS of the 21st century. Although elaborate structures with narrow and deep trenches can now be realized by state-of-the-art fabrication technologies, there always exists a strong need for a novel technology which is able to fabricate complex structures in a simpler way.

Narrow channels with high aspect ratios are frequently required for the high performance of a microscale system by increasing the electrostatic force between the system elements.[1] Such a structure, however, cannot be easily fabricated because it requires an etching technique of extremely high selectivity and anisotropy.[2] Two techniques are widely used at present: LIGA (German acronym for lithographie, galvanoformung, abformung)[3−5] and DRIE (deep reactive ion etching). Both techniques have their own merits and demerits.[6−11]

LIGA, combination of lithography, electroforming, and plastic molding, is the fabrication technology which can make microstructures with extremely high aspect ratios of more than 100 with almost no limitation in shape, though synchrotron radiation source is not readily available everywhere.

DRIE is another option to obtain structures with high aspect ratios. ASE (advanced silicon etching) technology developed by BOSCH and Surface Technology Systems is a representative example of this technology.[6−9] In this technique, substrates are etched by alternating etching and passivation for the protection of the sidewalls. Structures with high aspect ratios are obtained through the control of various parameters such as the time ratio of etching to passivation step, power, gas flow rate, and so forth. Structures with high aspect ratios can be obtained with smooth sidewalls at a lower cost than LIGA. However, ASE requires control of multiple operating parameters which change with the progress of etching.[6,12,13] Moreover, it is extremely difficult to simultaneously obtain structures with different aspect ratios on an identical substrate.[14,15]

It has been reported that microstructures could be fabricated by using anodic aluminum oxide (AAO) as a substrate. Photonic crystal circuit with 100−400 μm width and 100−300 μm height was reported.[16,17] Various gas sensors were developed taking advantage of the nanoporous structure of

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AAO substrates.[17,18] An AAO has vertically aligned parallel pores. The average pore diameter is in the range of ten to a few hundred nanometers and the aspect ratio of these pores goes easily to thousands. By taking advantage of this unique structure, an AAO can be a candidate for the substrate in the fabrication of complex microstructures.[19] A somewhat different approach was also reported to fabricate microstructures with AAO substrates through the sol−gel processing, patterning, and etching process.[20,21]

In this paper, we report the fabrication of microstructures with complex shapes and high aspect ratios by a single-step wet etching of AAO substrates. The structures thus fabricated can be used either as MEMS/NEMS devices or as molds for soft lithography.

Experimental Section

High-purity Al sheets (99.999%, 1-mm thickness) were degreased by ultrasonication in acetone and electropolished in a perchloric acid/ethanol (1:4, volume ratio) solution (7 °C, 20 V, 5∼10 min) to remove large surface irregularities. The distance between the electrodes was adjusted to be about 5 cm. Two-step anodization was carried out in a 0.3 M oxalic acid solution with a constant voltage of 40 V at 15 °C. Then, the AAO substrate was covered with the thin aluminum layer of 500-nm thickness by thermal evaporation. The lithographic process was done in the clean room. Photoresist (AZ 1512) was spin-coated at 4000 rpm on the substrate, and then patterns were generated using the photomask through UV exposure for 27 s. After photopolymerization, nonreacted photoresist was removed by rinsing for 1 min in an AZ developer CD30. The patterns were transferred to the aluminum layer by removing the unprotected area in an aluminum etching solution (H_3PO_4:CH_3COOH:HNO_3:H_2O = 80:5:5:10 by weight %) for 20 min. Finally, the AAO substrate was wet-etched in a 5 wt % H_3PO_4 solution at 20 °C for 8 h.

Results and Discussions

Figure 1 schematically shows each step of the fabrication process of an AAO substrate. Also shown in the same figure are the SEM images of the surface of the substrate at each step. The whole process consists of six steps: (1) electropolishing of the surface of an aluminum sheet, (2) two-step anodic oxidation of the aluminum sheet, (3) deposition of thin aluminum layer by thermal evaporation, (4) photoresist (PR) pattern formation by lithography, (5) transfer of the pattern to the thin aluminum layer by etching, and (6) fabrication of the high aspect ratio structures by wet etching of the AAO.

Electropolishing of the aluminum sheet is necessary to obtain a smooth surface because the pores proceed to the direction normal to the interface between the aluminum and oxalic acid solution during anodic oxidation. The rms roughness of the particular sample in Figure 1a is about 3 nm.

Parallel pores were obtained by the two-step anodization technique.[22] The SEM photograph of Figure 1b reveals that the pores are hexagonally aligned and the interpore distance is about 100 nm. The length of the pores is about 65 μm which can be measured from Figure 1f.

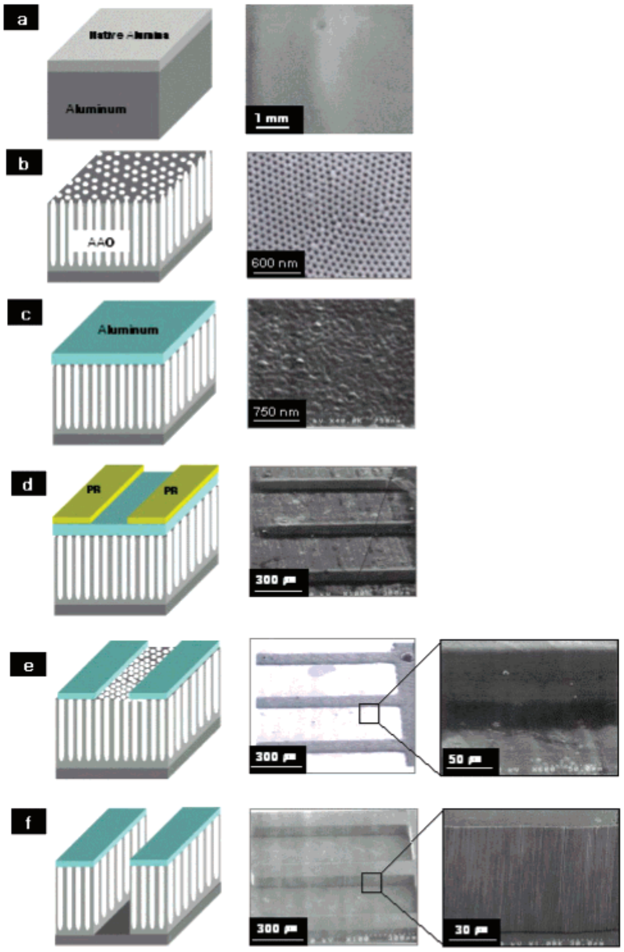

Figure 1. Overall fabrication process (a) electropolishing of the surface of an aluminum sheet, (b) two-step anodic oxidation of the aluminum sheet, (c) deposition of thin aluminum layer by thermal evaporation, (d) photoresist (PR) pattern formation by lithography, (e) transfer of the pattern to the thin aluminum layer by etching, and (f) fabrication of the high aspect ratio structures by wet etching of the AAO.

Figure 1c shows the surface of the thin aluminum layer deposited by thermal evaporation. This step is required to obtain a surface with high reflectivity which is necessary for successful lithography of elaborate and minute patterns. Such patterns are observed in Figure 1d.

The patterns were transferred to the underlying aluminum layer, the transfer layer, by immersing the sample into an etching solution. The aluminum etchant attacks and removes the area not covered by the PR, while the protected area remains unetched. The SEM photograph of Figure 1e shows that the patterns are faithfully transferred to the aluminum layer.

Finally, the microstructures were obtained from the AAO substrate by etching the sample in a reactive solution. Again, part of the AAO substrate which was exposed to the etching solution was removed, while the patterns which were protected by the aluminum transfer layer remained unetched. As a result, the three-dimensional microstructure with intended patterns was fabricated as shown in Figure 1f.

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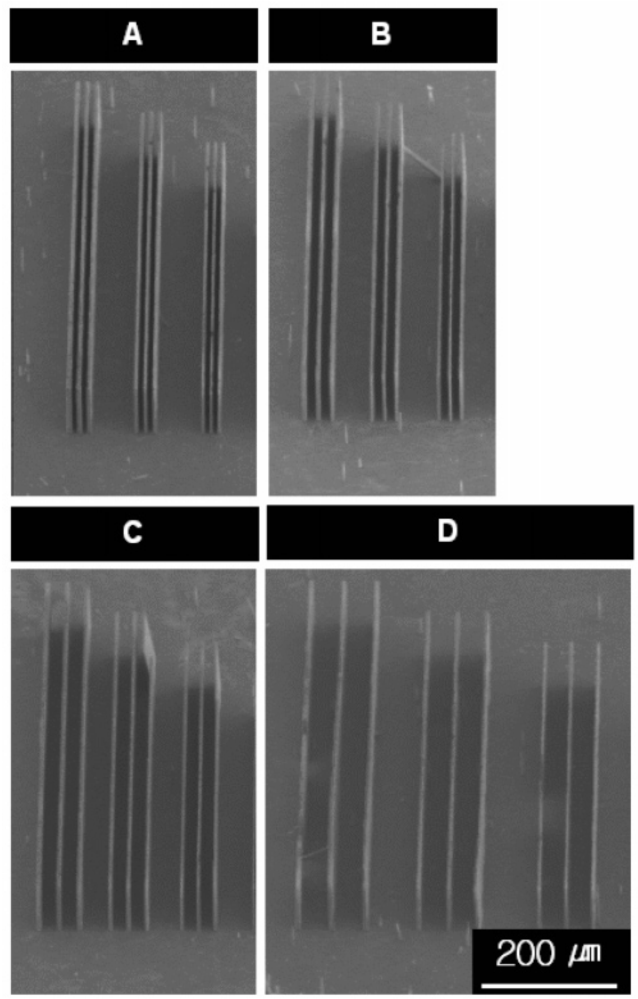

Figure 2. Simultaneous fabrication of the line structures with different width-to-gap ratios by the single-step wet etching of the AAO substrate. All of these structures reside on the same specimen. Width-to-gap ratio: (A) 2, (B) 1, (C) 0.5, and (D) 0.25.

Figure 2 shows the SEM photographs of the line structures with four different width-to-gap ratios. These structures were fabricated simultaneously by the single-step wet etching. All of them reside on the same specimen. The heights of these structures are identical everywhere. On the other hand, it is very difficult to achieve the same depths for the coexisting line structures with a wide range of width-to-gap ratios in DRIE.[9]

Another important feature of these structures is the vertical sidewall. In a typical wet-etching process with a silicon substrate, vertical sidewalls are hardly obtained because of the isotropic etching by an acid solution, resulting in severe undercut of the PR-protected structures.[23] The structures in Figure 2 show that undercut can be suppressed and high aspect ratio structures with vertical sidewalls are obtained by wet etching.

Anisotropic etching of an AAO substrate is made possible by the presence of the vertically aligned pores in the AAO. Figure 3 compares the difference between a conventional silicon wafer and an AAO substrate. An etching solution attacks the substrate from all directions in the case of a silicon wafer, resulting in rounded sidewalls. On the other hand, the etching solution fills the open pores of an AAO substrate, while the closed pores protected by the aluminum transfer layer remain empty inside (Figure 3b). Isotropic etching occurs inside the open pores of the AAO, and the walls of the open pores are dissolved and collapsed by the attack of an etching solution. The walls of the closed pores at the boundary of the patterns can also be dissolved by etching, though the chance of survival is high because of the etching from only one side compared with the etching of the walls of the open pores from both sides. A slight undercut does occur even with an AAO substrate, but the sidewalls of the structure can be vertical, and anisotropic etching can be achieved in a larger scale. The presence of vertical pillars in Figure 3c is the proof of lateral etching.

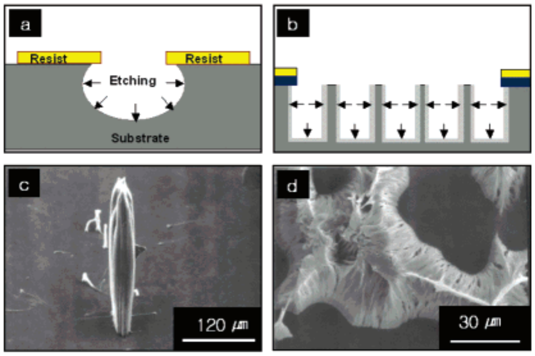

Figure 3. Different etching modes. (a) Isotropic etching of a Si substrate, (b) parallel etching of the pores of an AAO substrate, (c) vertical pillar due to the incomplete etching, and (d) collapse of the pore walls because of fast etching.

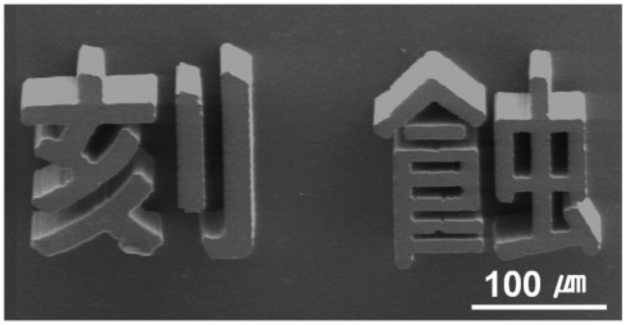

Figure 4. Fabrication of the complex structures which contain the parts of different aspect ratios.

Precision etching to obtain accurate structures depends on the balance between the dissolution of alumina walls and the diffusion of active etching components from the bulk solution into the pores. Ideally, slow etching is desirable because there is enough time for the diffusion of active etching components into the pores. Preferential etching of the pore mouths occurs with a faster etching rate. Figure 3d is an example of the fast etching, resulting in the collapse of the pore walls. Slow etching at low temperature, however, may take much longer fabrication time.

Structures with much complex patterns can be obtained by this method without any difficulty. Figure 4 shows the microstructures fabricated by a single-step wet etching. They are Chinese characters which form a Chinese word meaning “etching”. The narrowest width of the trenches in these structures is 3 μm, and the height is about 65 μm, so that

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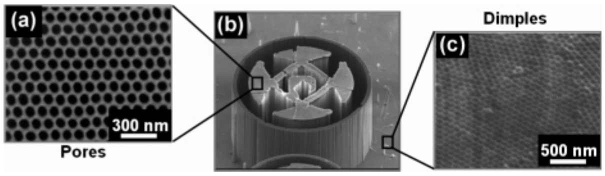

Figure 5. Tailoring of the surface morphology. (a) Pores, (b) micropattern of the symbol of the POSTECH, and (c) dimples.

the maximum aspect ratio of the trench reaches 22 in these structures. This figure demonstrates the ability to fabricate complex structures possessing different aspect ratios in a single-step wet etching. It is expected that trenches and holes of various aspect ratios can be obtained simultaneously by this method, which is a difficult task with conventional DRIE.

One interesting feature of AAO substrates is that surface morphology of a microstructure can be tailored. Figure 5 a is the SEM photograph of the surface, which is the symbol of POSTECH (Figure 5b), after the removal of the aluminum transfer layer. Nanosized pores are clearly visible in this figure, which is a characteristic of an AAO substrate. These pores provide a large surface area which can be used as a sensing surface, or they can be used as a template for replicas of metals or polymers. A smooth surface can also be obtained when we do not remove the aluminum transfer layer. On the other hand, nanodimples are present on the area of complete etching, where the aluminum substrate under the AAO is uncovered. This is shown in Figure 5c. A replica of this surface would have nanodimples of reverse curvature.

Conclusion

We have demonstrated the fabrication of complex structures with high aspect ratios by the single-step wet etching of AAO substrates. Structures with different shapes and aspect ratios can be obtained simultaneously with vertical sidewalls. Isotropic etching occurs only inside the open pores, which are not covered by the aluminum transfer layer, resulting in anisotropic etching of the AAO substrate. Precision etching to obtain accurate structures depends on the balance between the dissolution of alumina walls and the diffusion of active etching components from the bulk solution into the pores. The surface morphology of fabricated structures can be tailored. Since this fabrication technique is straightforward, versatile, and easy to be modified, it is expected that various MEMS/NEMS devices can be built using this technique.

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