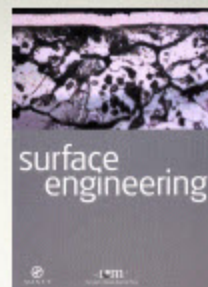


N Thin film silicon substrate formation using electrochemical anodic etching method

Authors: Kwon, J.-H.¹; Lee, S.-H.²; Ju, B.-K.³

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Abstract:

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Keywords: Porous silicon, Electrochemical etching, Anodisation, Layer transfer

Introduction

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Porous silicon can be formed by an electrochemical anodisation (ECA) method, as well as by a chemical etching (also known as stain etching) method using monocrystalline silicon (mono-Si). The chemical etching method is very simple because it requires no electrodes and no applied voltage; it produces PS on both sides of the Si wafer. Porous silicon formed by the chemical etching method is less steerable by external process variables⁷ than PS formed by the ECA method. With a given set of conditions, the chemical etching is self-regulating so that the simultaneous control of the PS layer parameters (thickness and porosity) is difficult to achieve. The ECA method is, however, the most extensively used so far. The structure of the resulting PS layer is strongly dependent upon the fabrication controlling technical parameters⁷ such as electrolyte composition, current density, etching time, etc. as well as the type of substrate used.

In this work, the authors describe a layer transfer (LT) technology⁸ based on the transfer on mono-Si films onto

a foreign substrate. Key processes in LT are double layered PS layers with different porosities formed by the ECA method owing to the splitting of the bonded pairs within the PS layers. Therefore, the authors study the PS formation is experimentally studied and analyse the double layered PS structure to transfer the thin film substrate as a byproduct of ECA.

Experimental

The starting material was a boron doped *p*-type mono-Si wafer (100) with resistivity ranging from 0.01 to 0.02 Ωcm and a sample area of $2 \times 2 \text{ cm}^2$. First, the mono-Si surface was cleaned by RCA (Radio Corporation of America) I and RCA II in boiled solutions ($\text{H}_2\text{O}/\text{H}_2\text{O}_2/\text{NH}_3\text{OH} = 7:1:1$) and ($\text{H}_2\text{O}/\text{H}_2\text{O}_2/\text{HCl} = 7:1:1$) respectively, and dipped into (5%) HF for several seconds to remove any native surface oxide and any residual contamination from the solutions.

Electrochemical anodisation was performed using a mixed solution (i.e. 1:1:1 volume ratio mixture of 49% HF solution, absolute EtOH (ethanol), and DI (deionised) water). The bath contained polytetrafluoroethylene (PTFE), and a Pt electrode was used as a cathode. A counterelectrode was used as a Si wafer/Pt. The principle of the equipment is shown schematically in Fig. 1. The bath was agitated by a magnetic stirring bar to prevent H_2 bubbling over the etching surface, so a more uniform PS structure was fabricated in the dark at room temperature ($\sim 23^\circ\text{C}$).

The double-layered PS of the separation layer was formed by changing the current density and the etching time. The first PS layer (start layer) was etched at a current density of 1.5 mA cm^{-2} for 10 min. Right after etching the first layer, the second layer (separation layer) was etched at a current density of 100 mA cm^{-2} for 10 s in the galvanostatic mode (WonATech WMPG 1000).

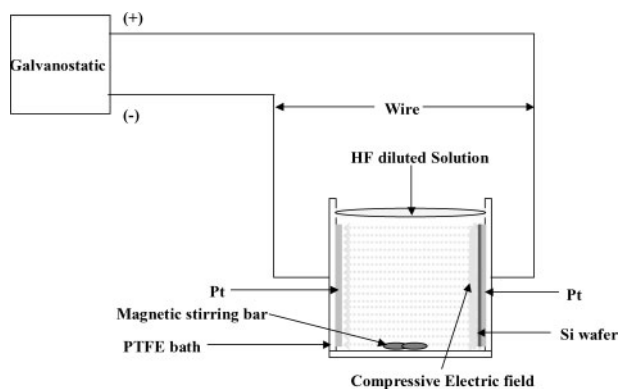
After PS formation, the samples were rinsed with DI water and dried immediately after ECA to prevent the PS film from flaking and deteriorating (i.e. the samples were dried in N_2 gas and stored).

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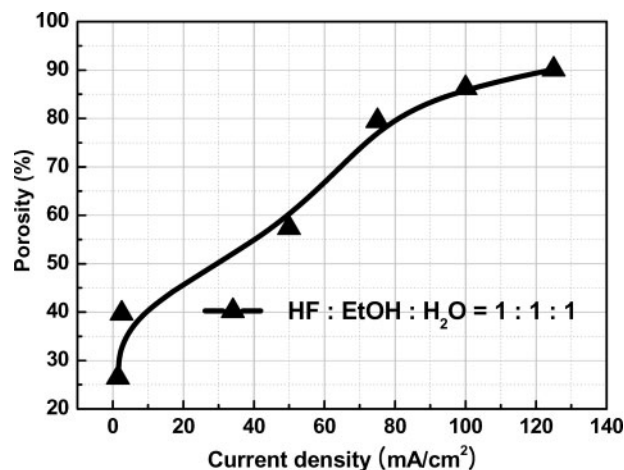
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Results and discussion

In the authors' work, PS layers of different thicknesses and porosities were formed on several samples by varying the etching time and current density, and the porosity of the PS layer was confirmed as shown in Fig. 2. In fact, at 1.5 and 2.5 mA cm⁻², the ECA duration is 600 s longer than at 50–125 mA cm⁻². In other words, there is almost no change of porosity when the time is relatively shorter in the ECA duration at low current densities than high current densities. Therefore, a PS layer was formed with an ECA duration of 600 s at the low current density (1.5 and 2.5 mA cm⁻²). However, other samples were formed by higher current densities (50–125 mA cm⁻²) for 60 s. When high current densities (>125 mA cm⁻²) are used, the porosity may not be confirmed at all, because the PS layer obtained by ECA is peeled off. A flaked and deteriorated PS film generated at 130 mA cm⁻² was observed. From these results, the authors determined the double layer PS conditions with a low current density and high current density at 1.5 and 100 mA cm⁻², for etching times of 10 min and 10 s respectively. The ECA of Si with the above conditions leads to the formation of a low porosity layer with a porosity of approximately 26% as calculated from the gravitational method.^{9,10} The etching rate is rather high, ~500 nm s⁻¹. At the depth of 3–4 μm from the surface, a high porosity (~86%) layer forms underneath the low porosity layer as shown in Fig. 3.

The double layered PS for LT that consists of two layers of different porosities can be easily created by increasing the current density in the middle of the ECA process. The first PS layer must have a low porosity so that the thin film can be transferred with quality to the foreign substrate; the porosity of this PS layer cannot be varied to improve the splitting. In other words, double layered PS formation for splitting needs to optimise the structure (i.e. porosity) of the second PS layer. In this way, the porosity of the second PS layer is an extremely important factor in the successful restriction of the splitting plane to be within a certain limited region.

Figure 3 shows the surface and cross-sectional FE-SEM images of as-anodised double layered PS for the LT process. The pores in the second PS were much larger than those in the first PS. In the first step, the first PS surface layers of ~3.2 μm in thickness with low

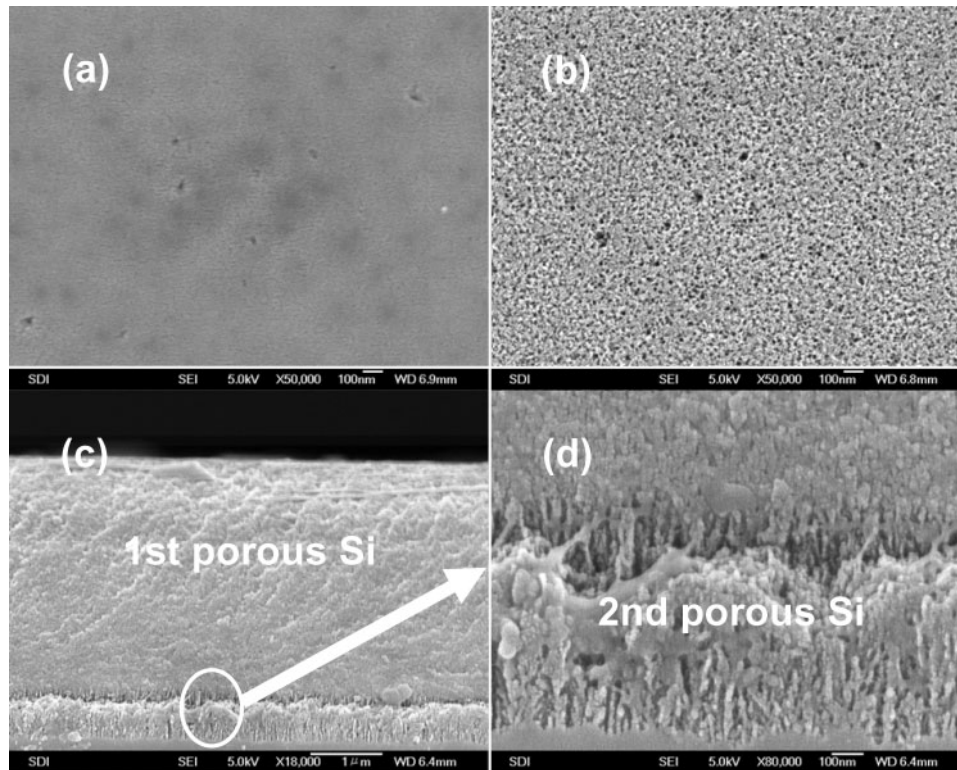


2 Porosity as function of current density for mono-Si wafer (100) substrate (0.01–0.02 Ωcm) and fixed ECA durations of 60 s at higher current density (>50 mA cm⁻²) and 600 s at low current density (<2.5 mA cm⁻²) using 49% HF/EtOH/H₂O solution

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Conclusions

In this work, the authors studied PS formation experimentally and demonstrated the formation of the separation layer located around the interface between the first and second PS layers. Electrochemically etched double layered PS layers of low porosity (26.5%) and high porosity (86.3%) were formed and processed at current densities of 1.5 and 100 mA cm⁻² for etching times of 10 min and 10 sec respectively in the galvanostatic mode. From these results, using double layered PS for LT is considered a viable technology to compete



3 Surface FE-SEM images of double layered PS formed as *a* first PS layer of low porosity, *b* second PS layer of high porosity, *c* cross-sectional FE-SEM image of double layered PS for LT process and *d* enlarged FE-SEM image of separation layer between first and second PS layers

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Acknowledgements

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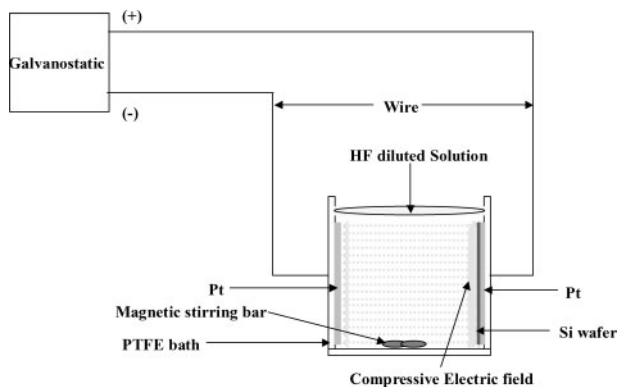
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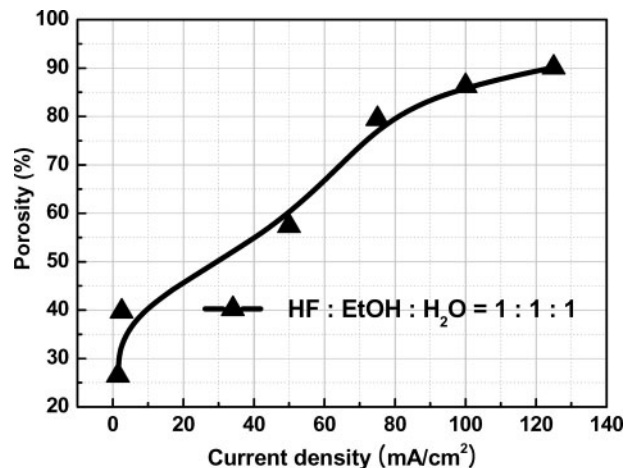
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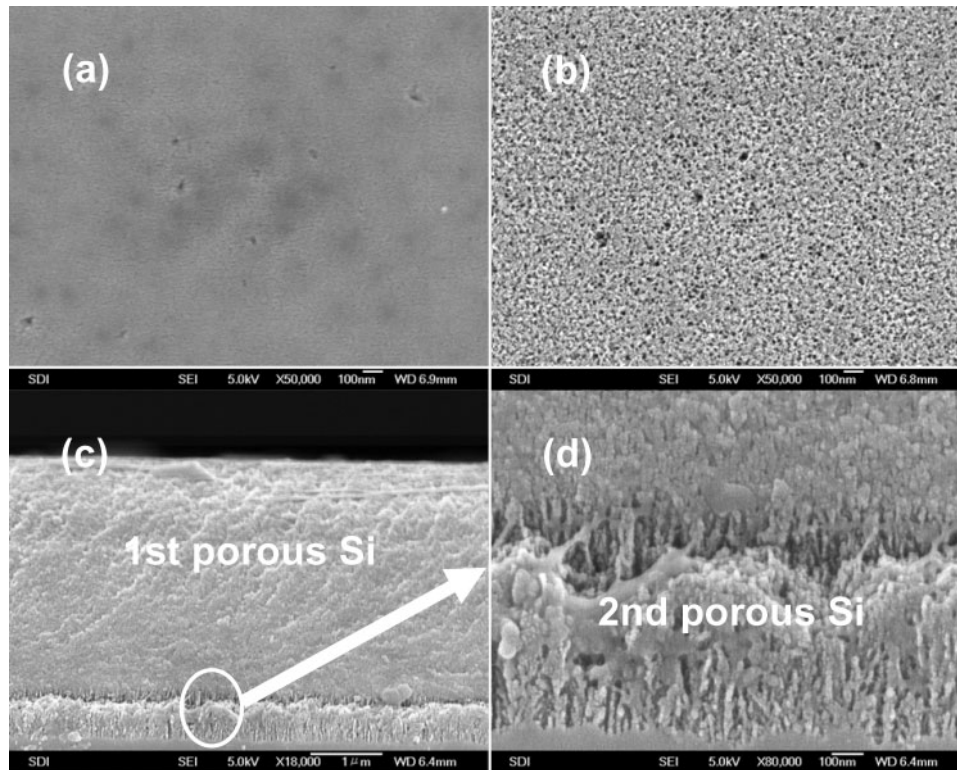


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