Self-Replication of Deeply Buried Doped Silicon Structures, which Remotely Control the Etching Process: A New Method for Forming a Silicon Pattern from the Bottom Up

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1. Introduction

Microstructuring (patterning) of silicon is one of the key steps in the manufacturing of integrated circuits (ICs), which, in turn, are keystones of modern electronics. Microstructuring is accomplished by a combination of procedures, which involve photolithography to create a template of the intended structure (mask), etching to remove the material under controlled conditions, and/or doping to incorporate foreign atoms into silicon, such as boron or phosphorous, by means of ion-implantation or diffusion through the mask.\(^{[1,2]}\)

A typical microstructuring process utilizes photolithographic masks to create arbitrary patterns on silicon substrates in a top-down approach. However, a new, bottom-up microstructuring method is reported, which enables the patterning of n-doped silicon substrates to be performed without the need for application of etch-masks or stencils during the etching process. Instead, the structuring process developed herein involves a simple alkaline etching performed under illumination and is remotely controlled by the p-doped micro-sized implants, buried beneath a homogeneous n-doped layer at depths of 0.25 to 1 \(\mu\)m. The microstructuring is realized because the buried implants act upon illumination as micro-sized photovoltaic cells, which generate a flux of electrons and increase the negative surface charge in areas above the implants. The locally increased surface charge causes a local protection of the native silicon oxide layer from alkaline etching, which ultimately leads to the microstructuring of the substrate. In this way, substrates having at their top a thick layer of homogeneously n-doped silicon can be structured, reducing the need for costly, time-consuming photolithography steps.

More precisely, microstructuring of silicon wafers is generally executed in five steps: 1) deposition of a homogeneous layer of mask material on the substrate, for example, a photosensitive polymer-based resist; 2) transfer of the pattern from the photomask to the photosensitive resist by light irradiation; 3) development of the photomask, that is, the removal of exposed (positive resist) or unexposed (negative resist) areas; 4) etching of silicon, typically performed by dry etching, wet anisotropic etching or wet isotropic etching; 5) removal of the etch mask, for example, by solvents.\(^{[11]}\) As modern ICs and microelectromechanical and micro-optoelectromechanical systems devices are highly complex, 3D structures consisting of a large number of structured layers, their fabrication necessitates the repetition of these steps multiple times for a single wafer, each time requiring precise alignment of the new mask with respect to the pattern created in a previous step. Such processes take up to 40% to 50% of the total wafer processing time, while the fabrication complexity makes the device susceptible to defects and contamination, which reduce production yield.\(^{[3–5]}\)

Attempts were undertaken to simplify the standard microstructuring process. One method utilizes a so called etch-stop effect.\(^{[6–8]}\) It was shown that the etch rate of a heavily boron-doped silicon is significantly reduced compared to undoped or n-doped silicon. However, high doping concentrations cause a transition of silicon from a semiconductor to a conductor,\(^{[1,9]}\) which disables its use for complementary-metal-oxide-semiconductor technology.\(^{[10]}\) Other attempts use varying mask materials and etchants,\(^{[15–14]}\) electrochemical etching,\(^{[15,16]}\) dry etching, and self-assembly of strained substrates\(^{[17]}\) or micro-contact printing techniques.\(^{[18]}\) Methods described in the patent literature use differences in photo-electrochemical properties of silicon doped in different ways.\(^{[19]}\) There, samples with a surface doping structure of p- and n-doped silicon can be selectively etched to remove only one type of silicon (either n-doped or p-doped), depending on the bias and illumination. Although these methods do not use masks during the etching process, they involve substrates having patterned surface heterogeneities created in previous steps.

Herein, we report an unprecedented bottom-up microstructuring process performed by means of photo-activated alkaline...
etching of substrates having no detectable features located directly at their surface but containing p-doped silicon features (further denoted as “buried implants”, p-Si) located deeply inside the n-doped silicon substrate, which provides remote control during the etching process (Figure 1).

An important difference between the method presented herein and previously reported methods, which make use of different reactivities of n-doped versus p-doped silicon against alkaline etchants, is that the literature methods utilize substrates having patterns of differently doped silicon areas incorporated directly into the topmost layer of the substrates. During the etching, these differently doped areas are then brought into a contact with the etchant. This stands in great contrast to our method, which deals with sub-areas are then brought into a contact with the etchant. This stands in great contrast to our method, which deals with sub-areas are then brought into a contact with the etchant.

Optimization of the etching parameters determined that a temperature of 50 °C, NH₄OH concentration of 0.6 wt% and illumination with a red LED light source (660 to 730 nm wavelength) are optimal conditions, enabling a reasonably high etch rate of ≈3 nm s⁻¹ and an excellent etching selectivity. Higher temperatures and concentrations led to a significant degradation in the resulting structure, due to a too high etch-rate. Conversely, a lower temperature led to a significant increase in process time without any gain in result quality. The optimized illumination wavelength can be rationalized with the known silicon absorption properties, suggesting an ideal absorption of light in the region of the buried implants at the given wavelength. Shorter wavelength photons, absorbed more strongly, cannot penetrate far enough, while photons of longer wavelength are not absorbed in the correct region or not at all if their energy is smaller than the silicon band gap. Accordingly, the

2. Results and Discussion

Fabrication of the starting substrates used in this work involves photolithography and ion implantation steps for the preparation of hexagonal arrays of hexagonally shaped p-doped silicon structures (d = 2–5 µm, Figure 1b) embedded into the topmost layer of the n-doped silicon (Figure 1a, n-Si). The fabrication process is finalized by epitaxial growth of 250 to 1000 nm thick layers of n-doped silicon (Figure 1a, n-Si). Atop all samples, a native oxide layer with a thickness of 1.4 nm is present. These substrates were used in the etching experiments, reported herein leading to the replication of the original pattern of the buried implants.

Prior to the etching experiments, the original samples were examined in order to detect any possible inhomogeneities that could potentially trigger the structuring process. Figure S1, Supporting Information, collects atomic force microscopy (AFM) topography, phase contrast, surface potential, and conductive mode AFM images, which do not disclose any features related to the expected pattern.

Basic anisotropic etching of the starting substrates performed in solutions of NH₄OH, TMAH (tetramethylammonium hydroxide), or KOH, occurs uniformly over the whole surface and does not produce any pattern, if performed in the dark. In contrast, etching in 0.6 wt% NH₄OH solution performed under illumination, produced by a 50 W halogen broadband lamp, results in hexagonal arrays of hexagonally-shaped pillars with their height controllable via etching time (Figure 2).

Superimposition of the formed patterns (Figure 2c) with the theoretical pattern of the buried p-doped implants (Figure 1), clearly demonstrates that the formed pattern replicates the original one, although the formed features are larger (by 0.5–0.8 µm at the top and ~2 µm at the base of the pillars, Figure 2b). Similar results were obtained with implants buried at a depth of 250 or 1000 nm (see Figure 2c and Figure 2e, respectively). The etch depth scaled with the top epitaxial layer thickness, that is, higher thickness led to higher columns, where the column size even exceeded the thickness of the top epitaxial layer. The size of the resulting microstructures is generally independent of the burying depth, with only samples with 1000 nm epitaxial layer thickness showing degradation in the roundness of the columns. Notably, the patterning was achieved uniformly over the whole sample surface, as demonstrated in Figure 2e.

Figure 1. Schematic representation of the starting substrates used for selective etching experiments: a) side view showing the layered structure, b) top view highlighting the hexagonal shape and orientation of the buried areas under the homogeneous n-Si and SiO₂ layers. The cross section shown in (a) is marked, as well.
optimized parameters were applied for all further experiments, if not noted otherwise. An increase of the etchant concentration speeds up the etching but reduces the selectivity.

The observed light sensitivity of the etching process points to a key role of the p-doped implants, which, being embedded into an n-doped silicon matrix, act as photovoltaic cells. In particular, we assume that pn-junctions, formed at the interface between the p-doped implants and surrounding n-doped silicon are responsible for the splitting of excitons that arise in the substrate upon absorption of light. The most actively operating pn-junctions (splitting the majority of carriers) are located closest to the illuminated etchant/substrate interface (marked by light-green areas in Figure 3). While photo-generated holes are injected inside the buried p-doped implants (blue arrows in Figure 3), most of the photo-generated electrons (yellow arrows) are injected into the n-doped region located above the implants. According to our hypothesis, these electrons, reaching the etchant/substrate interface, define the selectivity of the etching process. Two mechanisms for the influence of the photo-generated carriers are proposed (Figure 3). According to the first one, an ≈2 nm thick native silicon oxide layer on the surface of the substrate acts, in the presence of the buried p-doped implants, as a photo-responsive etching mask (Figure 3a). The second mechanism envisages a direct participation of photo-generated holes in the process of silicon etching (Figure 3b).

A series of experiments was performed to verify these hypotheses. In particular, time-dependent etching was carried out, which provided important information about the etching progression (Figure 4).

The height of the hexagonal pillars formed during the etching was evaluated by AFM and used as a quantitative characteristic of the etching progress. Two regimes were identified: i) an induction period, lasting around 300–400 s, during which no or little signs of etching were observed, and ii) the active etching period, characterized by an approximately linear dependence of the etch depth over time (see Figure S3, Supporting Information).

As follows from AFM-phase images (Figure 4e–h), the surface between the pillars becomes hydrophobic after etching, while the top surface of the pillars retains the hydrophilicity of the original samples. The high hydrophobicity of the etched regions is due to Si–H surface termination, whereas the hydrophilicity is induced by the native silicon oxide covering the surface. Topography images show that the regions between the pillars are much rougher (root mean square roughness, rms, of 29.9 nm), than the top surface of the pillars, as well as the original substrates (rms 6.7 nm). These results suggest that only areas outside the orthogonal projection of the buried implants to the surface undergo etching.

Figure 4a shows AFM images of the sample removed from the etching solution immediately after the time required for the etching of the native silicon oxide protective layer (330 s). Here, the pattern of the buried implants is replicated onto the surface with a height difference of only (1.8 ± 0.9) nm, which is comparable to the thickness of the native oxide layer. AFM phase image (Figure 4e), shows no or little material contrast, indicating that the native silicon oxide layer is not completely removed. This means that the etching has not yet progressed through the silicon oxide layer, but already caused a slight structuring. Obviously, the illumination induces a protective effect against the etching of the silicon oxide above the buried implants, whereas the surrounding areas undergo etching. After 20 s of active etch time, that is, the time after visible etching onset, (Figure 4b,f), the selective etching has progressed much further, leading to an etch depth of (38.5 ± 70) nm. With even further etch progress (+80 s: Figure 4c), the pattern is more clearly observed, and a maximum height of (401.8 ± 113.0) nm is reached after 180 s of active etch time. After 100 s of active etch time, the topmost surface of the columns starts to degrade as follows from increased roughness and decrease in the AFM phase contrast (shift to higher hydrophobicity). This indicates the onset of etching of the initially protected area in the orthogonal projection of the buried implants. The etching is accompanied by a degradation of the initial hexagonal shape toward a round shape and decrease in the column height. Thus, after 5 min of active etch time, the height of the pillars is only (187.4 ± 42.2) nm (Figure 4d). Overall, a near linear increase of the pillar height is observed for samples
with active etch time from 0 to 100 s, followed by a decrease of the height at longer etch times (Figure S3, Supporting Information). As seen from Figure S3, Supporting Information, the linear region corresponds to an etch rate of 2.98 nm s\(^{-1}\), which is somewhat lower than literature data at 8 nm s\(^{-1}\), characteristic for much higher etchant concentrations.[22]

In order to determine the resolution limit of the etching, the diameter of the structures formed upon etching were compared to the size of the corresponding buried implants. As seen from Figure S5, Supporting Information, the resulting structures are always larger than the corresponding implants and the difference (further named “enlargement”) decreases from \(\approx 800 \text{ nm}\) at 20 s to 100 nm at 100 s of the active etching time. This enlargement is most likely due to a random diffusion of the photo-generated electrons from the buried implants, which results in the expansion of the protected areas outside of the orthogonal projection of the buried implants to the surface (Figure 3).

The enlargement effectively defines the resolution limit of the etching – as soon as the distance between the buried features becomes equal or smaller than the doubled enlargement value, the protected areas induced by adjacent buried implant features merge, so that etching cannot occur in between.

The above-discussed experiments are consistent with the first mechanism hypothesis, illustrated in Figure 3a, which assumes a key role of the silicon oxide layer acting as an etching mask.

In general, silicon oxide (SiO\(_2\), silica) layers are widely used as masks for basic etching of silicon because the etch rate of silica is 3–4 orders of magnitude smaller than the etch rate of silicon.[6] The native silicon oxide layer displays a high stability in alkaline solutions because of ionizable groups (Si\(\equiv\)OH) present on the surface. In aqueous solutions, Si\(\equiv\)OH groups dissociate at pH > 2.6 to form Si\(\equiv\)O\(-\), creating a negative charge on the surface.[23] As the alkaline etching involves the nucleophilic attack of hydroxide anions (Equation 1), the negative surface charge repels the anions, slowing down the etch progress.[6,10]

\[
\text{Si} + 2\text{OH}^- + 2\text{H}_2\text{O} = \text{Si(OH)}_3^- + \text{H}_2\text{O}
\] (1)

An additional increase of the repulsive forces would then decrease the etch rate even further. We found that under standard etching conditions used in this work (50 °C, 0.6 wt% NH4OH), the dissolution of the 2 nm thick native silicon oxide layer under dark conditions takes about 6 to 7 min. The same induction period is needed in experiments performed under light irradiation. In this case, however, only areas outside the projection of the p-doped implants to the surface undergo etching (Figure 3a). Photo-generated electrons injected into the n-doped area negatively charge the surface areas above the p-doped implants, which further increases the negative charge on the native silicon oxide layer. This enhances the repulsion of hydroxide anions, preventing the etching. The passivation effect spreads only over areas just above the buried implants while in areas outside of the projection of the implants to the surface, the etching proceeds with a normal rate.

To further verify the proposed etching mechanism, photocurrent measurements were performed using a chronamperometry setup. In these experiments, two samples were investigated: the first sample type had no p-implants at all, while the second one had the p-implant layer extending over the entire sample, covered by a 1000 nm epitaxial n-Si layer (Figure 5). The back side of the sample was connected to the working electrode inside a special sample holder using Ga-In eutectic alloy to create an ohmic contact, and immersed in the etchant solution. In combination with a counter electrode and amperemeter, the current flow was measured in the dark and upon illumination by the previously employed LED light source. Figure 5 shows the results of the chronamperometry experiments performed at pH10 and pH2.

Under alkaline conditions, a sample without the buried implant shows a small, positive photocurrent, indicating that
upon illumination, electrons flow from the solution to the sample (Figure 5c, blue line). Conversely, the sample with the implant shows a strong negative photocurrent (Figure 5c, red line), indicating the flow of electrons from the sample into the electrolyte, which is in accordance with the first mechanism hypothesis for the etching selectivity (Figure 3a). Interestingly, the negative photocurrent decreases over the progress of the illumination and disappears completely at a certain point (after 200–250 s). This effect can be explained with photoelectrons accumulating at the surface and creating a barrier for the negative photocurrent, preventing further flow. On the other hand, this result suggests that the photo-generated electrons do not participate in redox reactions (e.g., reduction of the etchant) because otherwise a constant negative current would be observed upon illumination. If this explanation is valid, the photocurrent should be sensitive to any manipulations of the surface charge. To verify this assumption, the measurements were repeated at pH 2, at which the native surface charge of silicon oxide is expected to be positive due to the protonation of the surface Si–OH groups, in contrast to the negative surface charge at basic conditions. As seen from Figure 5d (red line), an order of magnitude increase of the initial negative photocurrent is observed at pH 2. This is in full agreement with the above-discussed surface charging effect, as the positively charged surface consumes more photo-generated electrons before a significant negative surface charge is built up, stopping the negative photocurrent flow.

To further verify the validity of the mechanism shown in Figure 3a, which assumes a key role of the native silicon oxide layer, etching experiments were carried out with substrates having no oxide layer. The oxide layer was removed in a 7:1 NH₄F:HF solution, which led to an increase in hydrophobicity of the substrate from <10° to ≈80° water contact angle due to the formation of Si–H terminal groups. The alkaline etching experiments were then performed under dark conditions and upon irradiation. In both cases, the active etching of silicon started immediately after the immersion of the substrates into the etching solutions, that is, without the induction period. Importantly, the etching proceeded homogenously over the full surface and no structuring was apparent in both cases.

We now consider the validity of the alternative mechanism hypothesis, which assumes that photo-generated holes escape from the p-doped implants, diffuse to the substrate–etchant interface and speed up the silicon etching process (Figure 3b). According to the mechanism, a part of the photo-generated holes, which are able to reach the substrate–etchant interface, generate interfacial silicon cations, which react with hydroxide anions from the etchant solution, producing water-soluble Si(OH)₂ species (Equation (1)). In this case, the areas above the buried implants are protected against the etching by the flux of the photo-generated electrons, whereas the areas outside the projection of the buried implants undergo accelerated etching. To verify the role of the illumination in the etching process, etching experiments were performed under illumination lasting for only ≈400 s (i.e., the time required for the etching of the silicon oxide layer). Afterward, the light source was turned off and the etching continued in the dark. Interestingly, the same patterned etch result was obtained in these experiments, as in the experiments performed under constant light illumination. Furthermore, after the protective film in the areas outside of the projection of the buried implant has been selectively removed during the 400 s etching under illumination, the subsequent etching in the dark continued (selectively in unprotected areas outside the projection of the buried implants) at the same rate as with the constant irradiation (3 nm s⁻¹). Thus, this experiment demonstrates that light plays the most significant role in the initial period, during which the differentiation of surface properties in different regions occurs due to selective etching of
silicon oxide, while the etching of silicon itself does not require the light irradiation. This result is consistent only with the first hypothesis of mechanism (Figure 3a), as the second considers the photo-generated holes as the main source of silicon etching and would require light illuminating during the whole etching process (Figure 3b).

3. Conclusion

In conclusion, a novel microstructuring process was developed for n-doped silicon substrates having buried p-doped implant areas in their internal structure. The process involves a simple alkaline etching performed under illumination and results in replication of the buried patterns. The microstructuring takes place because the buried implant structures provide a remote control during the etching process, acting as micro-sized photovoltaic cells, illumination of which produces a flux of photo-generated electrons directed from the buried implants toward the surface. Electrons accumulated at the surface increase the negative surface charge in areas above these implants. Locally increased surface charge causes a protection of the native silicon oxide layer against the alkaline etching in areas above the buried implants, which is responsible for the selectivity of the silicon etching. By looking at the mechanism underlying the developed process, we suggest that it falls to the definition of the bottom-up, rather than the top-down approach. Indeed, while in the standard top-down photolithography, the fabricated patterns replicate externally-applied lithographic masks or stencils; the microstructuring process reported herein is governed by the specific internal structure of the substrate and created patterns are formed by self-replication of the buried patterns. It should be however emphasized that the complex internal structure of the substrate was achieved by the standard top-down lithography, which is probably an unavoidable method when creation of arbitrary patterns is needed. The developed method is applicable only in cases when microstructures stacked on top of p-doped silicon imbedded into a matrix of n-doped silicon are targeted. Nevertheless, despite of its narrow scope, we believe that the developed process may have a significant and broad practical importance because it potentially reduces costly, time-consuming photolithography steps, providing a new tool for device manufacturing of stacked structures. Furthermore, we believe that the new microstructuring mechanism can inspire development of other processes, such as remotely-controlled, spatially resolved chemical reactions at the silicon interface. Work in this direction is currently underway.

4. Experimental Section

Sample Description: Substrates are silicon wafers with a socket epitaxial n-Si layer (4 × 10¹⁴ cm⁻³). Into the surface of this layer, hexagonal p-implants with sizes ranging between 2 and 5 µm and dopant concentration between 2 × 10¹⁵ and 1 × 10¹⁶ cm⁻³ are introduced by standard procedures (lithography, ion implantation). After implantation, another epitaxial n-Si layer (8 × 10¹⁴ cm⁻³) is deposited on top. The thickness of this layer was varied between 250, 500, and 1000 nm. The wafers were then cut into 20 × 10 mm sample pieces. For some experiments, samples without any implant or with full-area implant were used.

Sample Cleaning: Samples were cleaned in batches by a typical process, starting with ultrasonic washes in chloroform, acetone, and i-propanol. After rinsing and drying, samples were submitted to Standard Clean 1 (5:1 H₂O:H₂O₂:NH₄OH) at 60 °C for 20 min. The samples were removed from the cleaning solution and rinsed extensively with Millipore water.

Etching Procedure: Samples were then used immediately for etching experiments. For this, a sample was placed in a holder, designed to hold it vertical, and submerged in the etching solution in a quartz cuvette. For some experiments, a small magnetic stir bar was added. For each sample, new etching solution was prepared from preheated Millipore water with Millipore water.

For some experiments, a small magnetic stir bar was added. For each sample, new etching solution was prepared from preheated Millipore water and the alkaline etchant. The cuvette was then placed inside an aluminum holder with temperature control. Spaced 15 cm from the front side of the cuvette, a light source was placed. This was either a 50 W halogen household lamp or a LED light source with narrow wavelength distribution, fitted with a collimating lens (ThorLabs). The sample in the etchant solution was illuminated for 5 to 20 min, depending on the etchant and goal of experiment. Afterward, the sample was removed and rinsed extensively with Millipore water and dried in a N₂ stream. To investigate the time-dependence of the etching in dark conditions, where no formation of patterns was observed, a simple mask was created from
SU-8 photoresist by standard lithography techniques. The amount of etched silicon was then measured by AFM as the height of a step formed between the area exposed to the etchant and the area, which was covered by the resist during the etching (the measurements were performed after removal of the photoresist with the Standard Clean 1 procedure).

Supporting Information
Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest
The authors declare no conflict of interest.

Data Availability Statement
Research data are not shared.

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