Laser-Assisted Wet Etching of Silicon Back Surfaces Using 1552 nm Femtosecond Laser

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Abstract

Efficient three-dimensional (3D) microfabrication techniques of Si are in high demand for producing micrometer-scale 3D structures. Here, we report a new method for processing Si back surfaces using a 1552.5 nm femtosecond laser. As the Si is optically transparent at this wavelength, we attempted to machine the Si back surfaces via a nonlinear absorption process using the laser. Given that the etch rate of the back surface would be higher, wet etching was performed using an aqueous KOH solution. The 40% KOH solution was maintained in contact with the Si back surface at 25°C while the laser was irradiated from the front surface. The laser beam was focused on the back surface and linearly scanned under different conditions. Focusing the laser approximately 15 μm into the liquid yielded deeper grooves as compared to those when it was focused precisely on the Si back surface. Further, the etch rate was significantly higher compared to that during dry etching. We could achieve the maximum etch depth of approximately 6 μm during the wet etching process, in contrast to 0.3 μm during dry etching. However, the groove depth was not constant along the processing path. The results demonstrate a possibility of a new, efficient, and debris-free microfabrication technique.

Key words: Silicon, back surface, femtosecond laser, wet etching, potassium hydroxide solution

1. INTRODUCTION

Silicon is a key material in many modern devices such as electronic circuits, solar cells, and microelectromechanical systems, to name a few. Even though several techniques are available for the fabrication of these silicon-based devices, efficient three-dimensional (3D) microfabrication techniques in particular are in high demand, especially for producing micrometer-scale 3D structures [1-3].

Wet etching during laser irradiation is one such technique. The laser irradiation process can modify the rate of etching of Si by an etchant. Thus, by combining laser irradiation and treatment with an etchant, one can produce 3D structures on Si substrates. However, this process is a multistep one and usually takes a lot of time [4]. Further, while the etch rate of Si during laser-assisted wet etching is higher than that during etching in the absence of a laser, there are a few problems with the practical applicability of this method. For instance, the laser is usually irradiated onto the substrate through a liquid layer. This inevitably results in thermally induced bubbles near the position of irradiation. These phenomena can block or distort the incoming laser beam, resulting in unsteady and uncontrollable processing [5]. In addition, laser irradiation on a liquid layer perturbs the free liquid surface, resulting in the uncontrollable reflection/refraction of the incoming laser beam [6]. Furthermore, the debris produced tends to get deposited or suspended near the position or within the machined structure.

By performing wet etching on the back surfaces of the Si substrates, one can avoid these problems. Niino's group proposed laser-induced backside wet etching for the 3D machining of transparent substrates. In this process, a pulsed laser is irradiated onto the substrate and is absorbed by the liquid that is in contact with the back surface of the substrate; this produces a laser-induced plasma, which machines the substrate [7]. In addition, it is a debris-free process.

The 3D laser processing of transparent dielectric materials by short-pulse lasers via nonlinear absorption processes is also an established method for fabricating 3D structures on or within these materials [8-11]. We had shown previously that Si substrates are transparent to laser radiation at 1552.5 nm and that the laser processing of other materials located behind the Si substrate is possible using a pulsed laser. When pulses of a femtosecond 1552.5 nm laser are focused on materials such as Si, quartz crystals, or thin gold films through a single crystal Si substrate placed in front, the target can be processed through the Si substrate without causing any change in the Si substrate through which the laser is irradiated [12]. Thus, we can consider the Si substrate as being a "transparent" window at this wavelength. Based on these results, we attempted the 3D laser processing of both the interior and the backside of Si substrates using a femtosecond 1552.5 nm laser. While the preliminary results have been reported elsewhere [13, 14], we encountered difficulties during the experiments, which...
included a low machining rate and unsteady processing.

To achieve higher etch rates, we attempted the wet etching of the back sides of Si substrates by focusing a femtosecond 1552.5 nm laser onto the back surface of an Si substrate. The back surfaces of the Si substrates were maintained in contact with an etchant liquid. Thus, it was possible to avoid the problems encountered during conventional laser-assisted wet etching\(^\text{[15]}\). Liu et al. reported the processing of the back surfaces of Si wafers with nanosecond Nd:YAG laser pulses in air\(^\text{[16]}\). They reported the melting and transition of crystalline Si into oxidized and amorphous Si by heating via linear absorption of the laser radiation, because Si weakly absorbed the laser radiation of 1064 nm. Here, we report the results of further attempts at the wet etching of the back surfaces of Si substrates by irradiation with the pulses of a femtosecond 1552.5 nm laser through the substrates. The absorption of the laser radiation by Si is considered to be a non-linear process and thus more localized processing would occur.

2. EXPERIMENTAL

A schematic diagram of the laser irradiation system used is shown in Fig. 1. We employed an infrared (IR) femtosecond fiber laser (Discovery 1552-5, Raydiance) in this study. The irradiation system was operated at frequencies, \(f\), of 1 Hz to 500 kHz and pulse energies of 1 \(\mu\)J to 5 \(\mu\)J. The pulse width was 900 fs, and the central wavelength was 1552.5 nm. The laser pulses were passed through an IR microscope via the side arm using two mirrors and then focused. A visible-IR camera (ARTCAM-130MI-HDM-NIR, Artray) was installed along the observing optical axis. This setup made it possible to perform laser irradiation while observing the back surface of the test Si substrate using the IR camera. A dichroic mirror was installed inside the microscopy system such that the wavelength of incident laser radiation, 1552.5 nm, was reflected to an objective lens but that of the observed light, 700–1200 nm, was transmitted to an eyepiece. For the IR observations, we used an IR filter to observe the Si substrate using 1100 nm light.

An objective lens (\(\times 100\), numerical aperture of 0.85) equipped with a correction collar was used in order to minimize the aberrations arising from the high refractive index of Si (3.47 at 1552 nm\(^\text{[17]}\)). When a marking on the back surface of the Si substrate could be observed clearly after adjusting both the lens and the collection collar, it was assumed that the laser beam was focused on the back surface; this position was taken to be ±0 \(\mu\)m. Focus positions that lay below the back surface, that was in the liquid phase, were denoted using the minus (-) sign (e.g., -5 \(\mu\)m). Additional adjustments were made to the collection collar, given the slight difference in the refractive indices at the laser and observation wavelengths (3.47 at 1552 nm and 3.53 at 1100 nm\(^\text{[17,18]}\)).

Owing to the size of the laser beam and the acceptance diameter of the microscope as well as other losses, the measured energy of the laser pulses emitted from the objective lens was approximately one-fifth of that at the exit of the laser. In this study, we maintained the laser pulse energy at 4 \(\mu\)J, which reduced to approximately 0.8 \(\mu\)J after the objective lens.

Double-sided polished P-type Si substrates with a thickness of 320 \(\mu\)m and having the (100) crystal orientation were used in this study. The bandgap energy of single-crystal Si is 1.12 eV\(^\text{[19]}\). Further, the extinction coefficient of Si at the wavelength of 1552 nm is effectively zero. Hence, the Si is considered a transparent material in this IR region. Given that the etch rate at the Si back surface would be higher, wet etching using an aqueous KOH solution was performed. During the wet etching process, a 40% KOH solution at 25 °C was maintained in contact with the Si back surface, while the laser was irradiated from its front surface.

During trial experiments, we observed that bubbles formed during the irradiation process and that they prevented stable contact between the Si substrate surface and the KOH solution, resulting in nonuniform grooves along the processing path\(^\text{[15]}\). Therefore, we designed an irradiation cell, which is shown in Fig. 2. The Si substrate (20 mm \(\times\) 20 mm) was placed on the top of the liquid cell, in which the 40% KOH solution was circulated continuously using a pump. The back surface of the substrate was in complete contact with the etchant liquid such that the flowing liquid carried away the bubbles formed at the irradiated position. The sample cell could be moved using a motorized electric stage (ALS-602-H0M, Chuo Precision Industrial Co. Ltd.). During the scanning process, the sample was driven 500 \(\mu\)m in the \(x\)-direction and then moved 50 \(\mu\)m in the \(y\)-direction. After the completion of the irradiation process, the front and back surfaces of the sample were observed with an IR microscope, a laser microscope (VK-8710, KEYENCE), and a scanning electron microscopy (SEM) system.

Fig. 1 Schematic of experimental setup used in this study.
3. RESULTS AND DISCUSSIONS

3.1 Laser-Assisted Wet Etching on Si Back Surface with KOH Solution

Figure 3 shows optical micrographs of the Si back surfaces obtained at visible-range and IR wavelengths after wet etching during irradiation at \( f = 500 \text{ kHz} \); the scanning speed (\( v \)) was 50 \( \mu \text{m/s} \) and the focus position was at -15 \( \mu \text{m} \). We would like to emphasize here that there was no observable change on the front surface of the Si substrate, where the irradiated laser was incident. The laser scans appeared as black in the visible-light and IR images were the grooves etched on the back surface. In the visible-light images, only the changes that had occurred on the back surface could be observed. Thick black parts corresponded to deep grooves as shown in Fig. 3 while thinner straight lines were shallow grooves. Also, we did not observe any debris sticking around the processed lines as expected for a backside wet etching. This indicated that this method was a debris-free one. On the other hand, in the IR images, at some parts, slightly thicker gray lines appeared along the scan lines with thinner black parts. As reported in our dry-etching study, the gray parts observed with IR light are the changes occurring only inside Si. The IR images showed the changes that had occurred both on the back surface and within the substrate, where the incident laser was active. The pre-etch time in this case was 450 s. We measured the etch depth of the black area marked with the circle in Fig. 3 using a laser microscope; the pre-etch time in this case was 450 s. Figure 4 shows the depth profile along a length of 142 \( \mu \text{m} \) at the center of the black area. We considered the distance between the lowest part to the surface of the Si substrate to be the etch depth. The maximum groove depth was approximately 7.76 \( \mu \text{m} \). The depth varied significantly along the processing path, even though no bubble was observed on the back surface during irradiation either with a naked eye or using an IR camera. Moreover, even when the focus position, \( f \), and \( v \) were changed, similar variations were observed. The same variations in etch depth were observed for dry etching case but the absolute depth was much shallower than wet etching case.

An effect on the Si substrate even in the absence of laser irradiation. In the irradiation system used in this study, the Si back surface was in contact with the solution for some time prior to the laser irradiation process, and this contact period increased from the start position of the laser scan to its end. We defined the pre-etch time as the period for which the Si back surface and the KOH solution were in contact before the start of the laser irradiation process. In our experiments, the pre-etch time was 60 s, after which the irradiation process was started. Further, the pre-etch time increased from the top to the bottom for the scans performed in this order.

We measured the etch depth of the black area marked with the circle in Fig. 3 using a laser microscope; the pre-etch time in this case was 450 s. Figure 4 shows the depth profile along a length of 142 \( \mu \text{m} \) at the center of the black area. We considered the distance between the lowest part to the surface of the Si substrate to be the etch depth. The maximum groove depth was approximately 7.76 \( \mu \text{m} \). The depth varied significantly along the processing path, even though no bubble was observed on the back surface during irradiation either with a naked eye or using an IR camera. Moreover, even when the focus position, \( f \), and \( v \) were changed, similar variations were observed. The same variations in etch depth were observed for dry etching case but the absolute depth was much shallower than wet etching case.

### Table 1: Comparison of Etch Depth Between Wet and Dry Etching

| Scanning direction | Pre-etch time (s) | 100 \( \mu \text{m} \) |
|--------------------|-------------------|-----------------------|
|                    | 30                | 90                    |
|                    | 150               | 210                   |
|                    | 270               | 330                   |
|                    | 390               | 450                   |

**Fig. 3** Visible-light (left) and IR (right) microscopy images of Si back surface after wet etching at \( f = 500 \text{ kHz} \) and \( v = 50 \mu \text{m/s} \) at -15 \( \mu \text{m} \) focus position.

**Fig. 4** Depth profile of 142-\( \mu \text{m} \)-long section at center part of processing line for pre-etch time of 450 s. Position corresponds to circle in Fig. 3.

3.2 Effects of Focus Position
Figure 5 shows visible-light and IR images of the back surface subjected to wet etching at the same \( f \) and \( v \); however, in this case, the laser was irradiated at the 0 \( \mu m \) focus position. The lines in the visible-light image were narrower and fainter compared to those shown in Fig. 3. In addition, the lines were not continuous but intermittent, and there was no thick part, indicating that the etch depth was less than that in the case for a focus position of -15 \( \mu m \). In the IR image, the black dotted lines, which corresponded to those observed in the visible-light image, overlapped with wider, continuous gray bands. These gray bands represented the changes that occurred within the Si substrate and were not observed in the images in Fig. 3, which corresponded to a focus position of -15 \( \mu m \). The depth of the grooves formed at a focus position of 0 \( \mu m \) was approximately 300 nm and shallower than that of the grooves formed at -15 \( \mu m \) but greater than that of grooves formed by the dry etching of the back surface (approximately 150 nm)\(^{20}\). Thus, the focus position of -15 \( \mu m \) resulted in a higher etch rate (deeper grooves) compared to the 0 \( \mu m \) position. We tried other focus positions as well, namely, -12, -9, -6, -3, and +3 \( \mu m \). For long enough pre-etch time (360 s and longer), at the -12 \( \mu m \) focus position, we could achieve an etch depth of 8 \( \mu m \) at some points along the processing path. However, when the focus position was moved closer to the Si back surface, the maximum depth decreased. When we set the focus position to be within the Si substrate (+3 \( \mu m \)), the maximum etch depth on the Si back surface was very small, approximately 300 nm. Thus, it can be concluded that the laser must be focused at a point that lies beneath the etchant surface in order to increase the etch rate.

By performing laser-assisted wet etching on the back surfaces of Si substrates, we could form grooves with greater depths than that formed by dry etching. However, the depth of the grooves was not constant. For the same \( v \) and \( f \) values and focus position, the depth along the same processing path varied significantly from point to point. Even though the changes induced on the Si back surfaces were more uniform after the wet etching process than dry etching, we could not etch uniform and continuous deep grooves.

The etch rate at the -15 \( \mu m \) focus position was greater than that at the 0 \( \mu m \) position, even though the power density at the back surface was much lower in the former case. This contradictory result can be explained as follows. The optical absorption coefficient of water at 1552 nm is approximately 9.5 cm\(^{-1}\)\(^{21}\), and that of KOH solution is considered to be similar to that of water. Thus, owing to the high absorption coefficient of water, the aqueous KOH solution absorbs energy from the laser via a linear absorption process. Given the refraction of the laser light at the silicon–liquid interface, the focus position at -15 \( \mu m \) actually corresponded to a position 6.12 \( \mu m \) within the solution below the Si back surface under tight focusing. Thus, a small focal volume of the liquid close to the Si back surface absorbed a large amount of the laser energy, resulting in a rapid increase in its temperature. As a result, the etch rate of Si was high. At the 0 \( \mu m \) focus position, similar to dry etching, wherein nonlinear absorption occurred within the Si substrate near the focus. This decreased the amount of laser energy that reached the focus position\(^{20}\). Hence, the temperature of the KOH solution in contact with the surface was lower than that at -15 \( \mu m \), resulting in shallower depth.

![Fig. 5 Optical microscopy images of Si back surface obtained using visible (left) and IR (right) light after wet etching at \( f = 500 \) kHz and \( v = 50 \) \( \mu m/s \) at 0 \( \mu m \) focus position.](image)

### 3.3 Effects of Scan Speed \( v \) and Laser Repetition Rate \( f \)

Figure 6 shows microscopy images of the back surface of the Si substrate after it were subjected to wet etching at the 0 \( \mu m \) focus position at \( f = 500 \) kHz and different \( v \) values: 4 lines at 800 \( \mu m/s \), 4 lines at 400 \( \mu m/s \), 4 lines at 100 \( \mu m/s \), and 2 lines at 20 \( \mu m/s \). Although the pre-etch time inevitably increased from the top lines to the bottom ones, as those shown to Figs. 3 and 5, its effect was not evident in these images. We could form continuous grooves, with the contrast of the lines becoming sharper with a decrease in \( v \). At the lowest \( v \) value (20 \( \mu m/s \)), dotted lines were observed, suggesting that etching had occurred in a discontinuous manner. The average groove depth was approximately 400 nm and greater than that for \( v = 800 \mu m/s \).

![Fig. 6 Visible-light (left) and IR (right) images of Si back surface after wet etching at \( f = 500 \) kHz and different \( v \) at 0 \( \mu m \) focus position. From top to bottom: 4 lines at 800 \( \mu m/s \), 4 lines at 400 \( \mu m/s \), 4 lines at 100 \( \mu m/s \), and 2 lines at 20 \( \mu m/s \). Black rectangular lines are marking made by direct laser irradiation for adjusting focus.](image)
μm/s, which was approximately 200 nm. Continuous gray lines were seen in the IR images in addition to the black lines at the center, which corresponded to those observed in the visible-light images. The widths of the gray lines were greater than those of the lines observed in the visible-light images, indicating that the internal changes induced within the substrate had occurred over a wider area than those on the back surface.

3.4 Morphology on Back Surface after Wet Etching

We performed laser scans at $v = 50 \mu m/s$ at the 0 μm focus position with different $f$: 250 kHz, 125 kHz and 50 kHz. Even at the lowest $f$ value of 50 kHz, continuous etch lines were formed. Moreover, the average depth did not change significantly with different $f$ and remained at approximately 250–300 nm along each processed line. The internal changes observed in the IR images were continuous; however, in this case, the width decreased slightly with the decrease in $f$. Since the number of pulses irradiated at the same spot decreased with the decrease in $f$, the total amount of energy transmitted also decreased, resulting in a smaller internal modified area.

The focus position with different $f$ and $v$ values of 100 μm/s at the -15 μm focus position approximately 200 nm were observed; these were similar to the structures observed at the 0 μm focus position shown in (a). The depth of these periodic structures was low at approximately 200–300 nm. However, at the edge of the processing line, periodic structures parallel to the laser polarization direction were observed. Their interval was approximately 1.2–1.3 μm, and the depth was 0.8–1.5 μm.

After the wet etching process, we found so-called laser-induced periodic surface structures (LIPSS) or ripples at the bottom of the etched grooves. We believe this is the first instance where LIPSS were formed on the back surface of an Si substrate in an aqueous solution. There have only been a few studies on the formation of LIPSS at under water conditions, and in these previous studies, the LIPSS were formed on the front surface of the Si substrate under direct irradiation. Hence, the formation of LIPSS on the back surfaces of Si substrates in water is a unique achievement. The formation of LIPSS on materials surfaces is a subject of significant research interest, and a more detailed description of the LIPSS formation on the Si back surfaces can be found elsewhere.

4. CONCLUSIONS

In this study, we performed the laser-assisted wet etching of Si back surfaces using a femtosecond laser at 1552.5 nm. The etch rate on the back surface during the wet etching process was higher than dry etching, resulting in an increase in the groove depth from 300 nm to approximately 6 μm. We also examined the effects of laser repetition rate, the scanning speed and focus position on the wet etching process. A focus position approximately 12–15 μm below the Si back surface and within the KOH solution was found to be the most appropriate one for ensuring that the depth of the etched lines was the maximum. However, at the -15 μm focus position, which resulted in deeper grooves, the groove depth showed significant variations. At the 0 μm focus position, the depth was
shallower than -15 μm focus position but was deeper with better uniformity than that during dry etching. The morphology of the structural changes induced on the Si back surface by the laser-assisted wet etching method was different from that of the structures machined by the dry etching method[20]. In the former case, grooves and periodic structures either parallel or perpendicular to the laser polarization direction and having different depths were observed. The underlying formation mechanism of these structures will be elucidated in a future study. The results demonstrate a possibility of a new, efficient, and debris-free microfabrication technique.

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