Formation of Fine Periodic Structures on Back Surface of Si

Substrate by a Femtosecond Laser at 1552 nm

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Si is a semiconductor with a band gap energy of 1.12 eV, which corresponds to a wavelength of 1127.2 nm. Therefore, Si is considered as transparent for radiations longer than this wavelength. We have demonstrated that micromachining through an Si substrate by a femtosecond laser at 1552 nm is possible. We have found that periodic structures similar to the laser-induced periodic surface structure (LIPSS) with a periodicity of ~320 nm was formed on the back surface when the laser was focused on the back surface. The change of the surface was only observed on the back surface with no damage on the front surface. The LIPSS on the front surface were formed in perpendicular direction to the polarization plane of the laser irradiation conditions on the LIPSS have been studied. Furthermore, the LIPSS was found on the back surface after laser-assisted back-side wet etching by KOH solution, where the surface was in contact with water during irradiation. Both LSFL and HSFL were formed. Under some conditions, HFSL formed at center while LSFL formed on the edge of laser scan trace.

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1. Introduction

Silicon is an important semiconductor material underlying the modern technological society. Nowadays, as the semiconductor industry is developing quickly, there are large demands to find a method that can achieve high accuracy and precision machining of Si with low impact to the environment. The common Si processing techniques used widely in manufacturing IC, LSI and other electronic devices are based on the lithography. However, the lithography is essentially a 2D processing method using selective etching processes after patterning a resist layer and development of the layer. This is a complex process since it needs lots of chemicals, wash steps, patterning apparatuses and vacuum apparatuses etc. [1]. In order to provide a cost effective, efficient and reliable process, many technologies have been proposed to improve on the performance of Si micromachining [2-4].

Si is a semiconductor with band gap energy of 1.12 eV, which corresponds to the wavelength of 1127.2 nm. Therefore, Si is considered as transparent material for radiations longer than this wavelength. We have demonstrated that micromachining through an Si substrate by a femtosecond laser at a wavelength of 1552 nm is possible [5-7]. To increase the efficiency of Si processing, we proposed a new single step method for processing inside and on the back surface of Si substrates; non-linear processing inside and back surface of Si substrates using ultra-short pulse lasers at infrared wavelength [8-10]. During the course of the development, we have found that the periodic structures which have periodicity of ~ 320 nm is formed on the back surface of the Si substrate when the laser is focused on the back surface. These periodic structures seem quite similar to the well-known laser-induced periodic surface structure (LIPSS) or ripples, which are formed by short-pulse lasers on either front surface of absorbing materials or inside of transparent materials [11-15]. The LIPSS has attracted much interest because it provides single step surface structuring method and has wide practical applications. Most of the reported LIPSSs are formed perpendicular to the polarization. Recently, some reports describing the LIPSS formed in the parallel direction with the laser polarization have appeared. Also, two types of LIPSS, namely HSFL (high-spatial-frequency LIPSS) and LSFL (low-spatialfrequency LIPSS) have been reported [11-15]. LIPSS is also formed on the sidewall of machined structures such like trepanned holes on cemented tungsten carbide [16] or deep-hole drilling in copper [17]. The origin of these LIPSS is still under debates and some mechanisms have been proposed [18-27].



Fig.1 Schematic diagram of experimental set up [9].

The aim of this work is to report on the formation of the LIPSS on the back surface of an Si substrate by passing a femtosecond laser of 1552.5 nm through the substrate and to investigate the effect of the laser pulse energy and the laser repetition rate to the LIPSS formed on the back surface of the Si substrate. Also, the effects of laser polarization direction on the periodic structures are studied. The LIPSS is formed on the front surface by usual irradiation method and compared to those formed on the back surface. Formation of LIPSSs on a back surface of a substrate have rarely reported so far; those are our previous reports [9, 10, 28, 29] and one found during LIBWE [18]. The latter was found in peripheral of a spot irradiated by UV laser pulses on silica-pyrene/toluene interface, which were quite different conditions from ours. Furthermore, we have tried the laser assisted back-side wet etching of Si by KOH solution to enhance the machining rate on Si back surface [8, 10] and found that the LIPSS were formed on the back surface of the Si during the wet etching by KOH solution. Characteristics of these LIPSSs were investigated by a scanning electron microscope (SEM), an atomic force microscope (AFM) and a laser microscope.

2. Experimental

An infrared femtosecond laser system, which was operated at repetition rate, f, of 1 to 500 kHz and the maximum pulse energy of 5 µJ with a central wavelength of 1552.5 nm and pulse width of 900 fs was used (Raydiance Discovery 1552-5). A schematic diagram of the laser irradiation system is shown in Fig.1. The laser was steered by two mirrors, and then was transferred to an infrared microscope from the side of it. Inside the microscope, a dichroic mirror, which reflected the processing laser light at 1552 nm and transmitted the observing light at 1100 \sim 1300 nm, was installed. The laser was introduced via a side arm of the microscope and reflected to the coaxial direction with the observation optical system. A visible-infrared camera (Artray ARTCAM-130MI-HDM-NIR) was installed along the observation axis. For the IR observation, we used a filter to observe the Si sample using 1100-nm light. By this system, it was possible to carry out the laser irradiation while observing the back surface of the Si by the infrared camera [9].

An objective lens (x100, N.A. 0.85) equipped with a correction collar was used in order to minimize the aberration due to the high refractive index of Si. Since the laser beam diameter was 5 mm while the aperture diameter of the objective lens was 3 mm, the laser energy decreased while transmitting through the objective lens. Thus, with other losses of optical systems, the energy after transmitting through the objective lens was about 20% of the energy before the transmission. The pulse energy of the laser was evaluated by measuring the power of the laser light emitted from the objective lens, then divided by the repetition rate f. Thus, when we operated the laser at 4 μ J output, the pulse energy impinged on the Si substrate was approximately 0.8 µJ, which was further reduced by other losses such as reflection by optics and the sample interfaces before reaching the focus.

The sample used in the experiment was a double-sided polished P-type Si wafer. The sample had a thickness of 306 μ m having the (100) crystal orientation. A piece of 20 x 20 mm² wafer was fixed on a rectangular frame on a stage so that the back surface was an Si-air interface. The focus position was usually set on the back surface of the substrate which was denoted as the focus position of 0 μ m. Procedure of adjusting the focus position was described in



Fig.2 SEM images of periodic structures formed on the rear surface of Si substrates on the irradiated area by $0.8 \mu J$ laser pulses with repitition rates of (b) 400 Hz, (c) 1 kHz, (d) 10 kHz, and (e) 40 kHz. As shown in (a), any damage is not observed on the front surface of Si substrate where the changes shown in (b) to (e) are formed at the rear surface.

[9]. The refractive index, n, of Si is 3.53 at the wavelength of 1120 nm while it is 3.48 at 1552.5 nm [30, 31]. The correction collar compensated this difference of n at irradiated and observed wavelengths. We scanned the laser linearly to fabricate a groove, which was the simplest structure machined on the surface. The sample was moved using a motorized stage with a speed v. Electric stages used were ALS-602-HOM (Chuo Precision Industrial Co. Ltd.). In the scanning process, the sample was driven for 100 µm/s in x-direction, and then moved 50 µm in y-direction. An infrared microscope, a laser microscope, a scanning electron microscope (SEM), and an atomic force microscope (AFM) observed the morphologies of the back surface and the front surface of the sample after irradiation.

In wet etching experiment, a flow of 40 % KOH solution of 25 Celsius was kept in contact with the back surface of the Si substrate during the laser irradiation. In this case, the focus position was varied from 0 to -15 μ m, where – sign indicated that the focus was moved from the back surface to inside the solution by an indicated distance. Details were presented elsewhere [10].

3. Results

3.1 Formation of periodic structures on Si back surface

Fig. 2 shows SEM images of the periodic structures generated on the back surface of the Si substrate at v of 100 µm/s and f from 400 Hz to 40 kHz with pulse energy of 0.8 µJ/pulse. Fig. 2 (a) shows the SEM image of the front surface where the laser impinged. There was no noticeable change. Processed lines were generated on the back surface and they corresponded to the irradiation position of the laser. Laser-induced morphology changes on the Si back surface generally showed their dependence to the repetition rate f of the laser, as shown in (b) to (e). On the other hand, no damage was found on the front surface of the Si substrate for all f as shown in (a). These results show once again that it is possible to make position-selective processing on the Si substrate using the infrared femtosecond laser at 1552.5 nm. The periodic structure started to be observed at f = 400 Hz. It was found in the central part of the irradiated region. With the increase of the f to 1 kHz, the periodic structures became well organized and distinctive with the average period Λ of ~320 nm, which was ~ 1/5 to the laser wavelength ($\lambda = 1552.5$ nm).

Keeping the energy at 0.8 µJ/pulse, the characteristic evolution of the periodic structures was also observed with increasing the f to 4 kHz. It resulted in modifying the periodic structure to fine particle like structure at some parts of processing line. When the f was increased to 10 kHz or above at the same laser pulse energy, the morphology gradually changed from the periodic structures into the fine particle like structures at the center (d) and they completely covered the processed area for f = 40 kHz (e). Figure 3 shows changes of the Λ of the LIPSS on the back surface with laser pulse energy and repetition rate. The Λ was evaluated by measuring a distance between two neighboring periodic structures from SEM images. The Λ is between 260 nm and 340 nm in the range of the f from 400 Hz to 10 kHz. There is no big difference in the Λ formed under different pulse energy conditions, even though they have rather large standard deviations and decreased a bit



Fig.3 Periodicity Λ of fine structures (LIPSS) on the back surface as a function of the laser repetition rate *f* for the pulse energy of $0.2 \sim 0.8 \mu$ J/pulse.

with the increase in the *f*. The wavelength of the laser would become $\lambda_{Si} = \lambda/n = 1552/3.48 = 446$ nm in inside of the bulk Si, where n = 3.48 is the refractive index of Si at 1552 nm [30]. If we adopt this λ_{Si} to the laser wavelength relevant to the LIPSS formation on the back surface, the Λ is approximately $0.76 \sim 0.58$ times λ_{Si} , which coincide with an expected value of Λ for the normal LFSL on the front surface on many substrates. However, orientation of the LIPSS on the back surface is parallel to the polarization of the laser in contrast to the perpendicular direction in the normal LSFL [13, 15].

AFM was used to observe the details of the structure. AFM height images and the cross-section of the scanning area are shown in Fig. 4 (a) and (b) for the laser pulse energy of 0.8 μ J and a frequency *f* of 1 kHz. The depth of the periodic structures was found approximately 5 to 25 nm for every laser pulse energy condition.



Fig. 4 (a) AFM image and (b) corresponding cross section of periodic structures fabricated on the back surface at the laser pulse energy of $0.8 \mu J$ and the repetition rate of 1 kHz.

3.2 Formation of periodic structures on Si front surface

In order to compare with the LIPSS formed on the Si back surface to those on the front surface, the similar experiments were carried out on the front surface of the Si substrate. The morphology change on the front surface of Si is presented in Fig.5 (a) and (b). Laser irradiation was carried out at $v = 100 \mu m/s$ by setting the focus position at 8 µm above the front surface of the Si substrate. The focal position was moved upward from the front surface in order to get lower energy fluence. The f was varied in the range of 100 Hz \sim 500 kHz with constant laser pulse energy of 0.8 µJ. Processed positions were observed using the SEM. Fig.5 (a) and (b) show the SEM images of the morphology on the front surface of Si at the f of 1 kHz (a) and 100 kHz (b). Fig.5 (a) shows the LIPSS formed in perpendicular direction to laser polarization while (b) shows a groove with granular structures at the bottom and any ripple structure is not observed in it. The LIPSS started to appear when the f was 400 Hz. Then, the well-organized LIPSS was observed until 8 kHz. However, it started to collapse above 10 kHz and fine granular structures were gradually appeared and completely cover the laser trace for f higher than 20 kHz. The Λ was measured from the SEM images. 10 locations in the periodic structure of each image were picked up for measurement. Fig.6 shows the Λ of the LIPPS on the front surface as functions of the f. It was not affected by the f so long as the LIPSS formed and was approximately 1000 ~ 1150 nm. This periodicity was approximately 0.8 x λ and its direction was perpendicular to the laser polarization. These features are similar to the com-



Fig.5. SEM images of LIPSS on the front surface of the Si substrate after femtosecond laser irradiation with (a) 1 kHz and (b) 100 kHz repetition rate.



Fig.6 The graph shows the periodicity Λ of the LIPSS formed on the front surface as a function of the repetition rate *f* at constant laser energy of 0.8 μ J.

monly reported LIPSS (LSFL) on the materials surfaces [11-15]. The laser microscope measured the depth of the LIPSS formed on processed lines. The depth measurement was performed at 10 locations for each repetition rate. The depth was about $0.3 \sim 0.4 \ \mu\text{m}$ in the *f* from 400 Hz to 5 kHz. Then, the depth gradually increased, and it was found to be about 1.8 $\ \mu\text{m}$ when the *f* was 20 kHz. The periodicity and the direction of the LIPSSs formed on the back surfaces and the front surface were not the same, suggesting the different mechanisms for their formation.

3.3 Effects of laser polarization on periodic structures

The direction of the periodic structures formed on the back surface was parallel to the direction of the laser polarization, while it was perpendicular for that on the front surface. To clarify the relation between the directions of frontand back-surface LIPSS and the laser polarization direction, we changed the direction of the polarization by inserting a half-wave plate into the laser beam path. It was changed as 90, 45 and 0 degrees against the laser scan path. Laser was irradiated on both the front and the back surface of 303 µm thick Si substrate at the v of 100 μ m/s and the f of 1 kHz. The pulse energy was 0.8 µJ. In irradiating the Si front surface, the focal position was placed at 10 µm above the front surface to reduce the fluence. Fig.7 and Fig.8 show the SEM images of processing lines on the back surface and the front surface of the Si substrate in the different directions of the laser polarization against the scan direction,

| Angle against Scan Direc- tion/Degree | | 90 | | 45 | | 0 |
|---|------|-------------|------|-------|------|-------|
| Scan direction | ţ, | Polarizatio | n K | | | |
| Width/µm | 12.4 | (0.1) | 12.0 | (0.2) | 10.8 | (0.2) |
| Periodicity Λ / nm | 360 | (20) | 370 | (40) | 400 | (60) |

Fig. 7 SEM images of the LIPSS on the back surface of the Si substrate for different polarization directions at 90, 45 and 0 degrees against the laser polarization direction (0.8 μ J/pulse, 1 kHz). Red arrow indicates scan direction and white arrow indicates laser polarization direction.

| Angle against | | | | | | |
|----------------------------|------|-------|------|-------|------|-------|
| Scan Direc- | | 90 | | 45 | | 0 |
| tion/Degree | | | | | | |
| Scanning direction | Į. | | | | | |
| Width/µm | 20.3 | (0.3) | 14.8 | (0.3) | 13.8 | (0.3) |
| Periodicity Λ / nm | 1100 | (200) | 1080 | (140) | 1000 | (100) |

Fig. 8 SEM images of the LIPSS on the front surface of the Si substrate for different polarization directions at 90, 45 and 0 degrees against the laser polarization direction (0.8 μ J/pulse, 1 kHz). Red arrow indicates scan direction and white arrow indicates laser polarization direction.

respectively. The width of the formed LIPSSs and their periodicity were measured at 10 places in each scan path. The average values are listed in the figures (tables) 7 and 8, where the parentheses represent the standard deviation.

LIPSSs were formed on both the front and back surfaces and their directions rotated by changing the laser beam polarization. The periodic structures on the rear surface were always formed in the direction parallel to the laser polarization and Λ was approximately 380 nm. When the plane of the laser polarization was perpendicular to the scanning direction, the width of the LIPSS was approximately 10% wider than that for parallel to the scanning direction, as shown in Fig. 7. On the other hand, the LIPSS formed on the front surface was always found to be perpendicular to the polarization direction. The Λ was about 1050 nm. When the laser polarization was perpendicular to the scanning direction, the width of LIPSSs was approximately 30% bigger than parallel cases, as indicated in Fig. 8. Thus, the LIPSS on the back and front surfaces were formed in contradictory direction against the laser polarization. The Λ of LIPSS on the back was approximately one third of that on the front. This difference would be explained by considering the difference of refractive index for air and silicon, namely 1.01 and 3.48, as discussed in the previous section.

3.4 Periodic structures found on the Si back surface after wet etching by KOH solution

We have carried out a laser assisted wet etching on the back surface of the Si substrate using KOH solution to increase the machining rate in laser processing on the back surface than dry, direct ablation [8-10]. After etching grooves on the back surface, the LIPSSs were formed under some laser irradiation conditions. The laser scan speed v and the repetition rate f employed in the wet etching were different to those in sections 3.1 to 3.3 to get higher wet etching rate; v from 50 µm/s to 400 µm/s and f from 125 kHz to 500 kHz [10].



After the laser-assisted backside wet etching at v = 50

Fig. 9 SEM images of LIPSS formed on the Si back surface after laser-assisted wet etching by KOH solution at f = 500 kHz and $v = 50 \mu$ m/s at (a) -15 μ m and (b) 0 μ m focus positions and (c) at $v = 100 \mu$ m/s at -15 μ m focus position. (d) shows the center region of figure (c).

to 400 μ m/s and 0 μ m or -15 μ m focus position, two types of morphologies were observed at the trace of the scan on the back surface by SEM observation; the grooves and the periodic structures oriented perpendicular or parallel to the laser polarization. Fig. 9 shows the SEM observations of the processed lines after wet etching with f = 500 kHz and $v = 50 \ \mu m/s$ at focus positions of -15 μm (a) and 0 μm (b). At the -15 µm focus position, the focus located at the inside of KOH solution, some parts of the processed line became the periodic structures directed parallel to the laser polarization with the Λ of approximately 1.2 μ m and the depth of 1.3 µm as shown in (a), which seemed like the LSFL. Some parts of the line became deep grooves with the depth up to 5 µm without the periodic structures at the bottom. At 0 µm focus position, the morphology on the back surface was dominated by periodic structures directed perpendicular to the laser polarization as shown in (b), which seemed like HSFL. The Λ was approximately 200 nm and the depth were between 170 to 300 nm. For f = 250 kHz or lower, the periodic structure was dominated while the trace contained both the grooves and the periodic structures for f = 500 kHz. It is different from the periodic structures in the case of the dry etching in which the Λ is approximately 320 nm and is directed parallel to the laser polarization [9]. Meanwhile, Figure 9 (c) shows the SEM observation of one processed line after wet etching with f = 500 kHz at v = 100 µm/s, a higher speed than (a), at -15 µm focus position. At the center of the groove, the periodic structures directed perpendicular to the laser polarization with Λ approximately 200 nm, which was quite similar to the structures observed after wet etching at 0 µm focus position (b), were formed as shown in magnified scale in (d). The depth of these periodic structures is very shallow, around 200-300 nm. However, on the edge of the processed line, the periodic structures parallel to the laser polarization direction, similar to those in (a), were observed. Its Λ was approximately 1.2-1.3 μ m and the depth was between $0.8-1.5 \ \mu m$.

4. Discussions

The periodic structures on the back surface of the Si substrate are very similar to the LIPSS that commonly formed on the surface of many substrates. In many cases, when ultra-short pulse lasers are irradiating on an Si or other solid surfaces, the LIPSS with an interval equal to laser wavelength or slightly shorter, is formed perpendicular to the laser polarization direction [11-15]. Formation of the LIPSS irradiated by IR wavelengths ($\lambda = 1300$ nm, 2100 nm) is reported for Si substrate [20]. Only the LSFL with periodicity Λ of 1050 nm and 1600 nm were formed when λ were 1300 nm and 2100 nm, respectively. Reports on the LIPSS with spatial period much smaller than the laser wavelength on compound semiconductors have been published [32]. The LIPSS with periodicity of 200 nm, which is approximately 1/4 to the incident laser wavelength $(\lambda = 800 \text{ nm})$, has been reported for crystalline silicon (100) surface under ultra-high vacuum [12, 19]. The direction of the HSFL is parallel to the laser polarization direction. The characteristics of the LIPSS showed by F. Costache et al is quite similar to our result [24]. However, the LIPSS, which have been reported so far, is formed by the laser irradiation on the front surface of substrate.

The periodic structures on the back surface of the Si substrate by irradiation of the femtosecond laser at 1552 nm through the substrate itself have only reported by us so far [9, 10, 28, 29]. Therefore, the mechanism of this structure formation has not known yet. In our study, the periodicity of structures Λ on the back surface of Si substrate was approximately 320 nm, which was $\sim 1/5$ to the laser wavelength. R. Buividas et al [33, 34] found that the Λ is close to the values of $(\lambda/n)/2$ where the λ/n is the wavelength in the material. Considering that the laser beam was irradiated through the Si substrate, the refractive index n of which is 3.48, the laser wavelength in inside of Si should become 1552 nm / 3.48 = 446 nm. This value was larger than $(\lambda/n)/2$ but ~ 0.76 (λ/n) , which was within the variation of Λ reported for the LSFL [15]. Therefore, we consider that the periodic structures that formed on the back surface supposedly have the Λ that is almost like the LSFL governed by the wavelength in inside the Si substrate. From Fig. 2 and Fig. 5 (a) (b), both periodic structures on the back surface and the front surface of Si substrate changed into fine granular structures in higher laser repetition rates. It is probable that, when the *f* increases, the diffusion of heat is not yet completed before the arrival of next laser pulse. Therefore, the temperature becomes higher, which makes both the absorption coefficient of Si and the multi-photon absorption rate to increase. We think that the morphology of periodic structure changes into the granular structures due to the temperature rise.

The LIPSSs formed on the back surfaces are quite unique and only a few have been reported [9, 10, 18, 28, 29]. Bohme reported the LIPSS formation on the back surface of fused silica in the LIBWE using an organic solvent by spot irradiation of 600 fs UV laser pulses [18]. The LIPSS was found at hot spots of ring structures in laserirradiated area. They suggested the contribution of the deposition of decomposition products (mainly carbon), which was not relevant in our case, for there was no carbon source.

After the wet etching of the back surface of the Si substrate, the LIPSS was formed on the bottom of the processed grooves. We think that this is the first report of the LIPSS formed on the back surface of a substrate in a solution (water). Several studies have been published on the LIPSS formed under water. These LIPSSs are formed by direct irradiations on the front surface of a metal or silicon under water layer. Effects of water or liquid environments have been discussed but their results do not always coincide with each other; sometimes contradicted results have been reported [21, 35-41]. Most of LIPSSs made on the front surface under liquid are the LSFLs formed in the direction perpendicular to the laser polarization direction. In some cases, the HSFL is reported to be formed in the parallel direction. Usually the LSFL is formed in perpendicular direction to the laser polarization direction [13, 15].

However, the LSFL formed on the back surface of the Si directed parallel to the laser polarization. Further, the HFSL is formed in the parallel or perpendicular directions depending on the laser irradiation conditions. What determines the direction of the LIPSS is still unclear. It is indicated that the HSFL is formed at lower laser fluences in comparison to the LSFL [15]. In the Fig. 9 (c) and (d), the HSFL is observed at the center of the processed line where

one expects higher laser fluence and the LSFL is observed on the edge of the line where the fluence should be lower than at the center. This paradoxical result suggests that the laser energy reaching at the central part would decrease probably due to the energy loss along its path induced by temperature increase or non-linear effects [9].

5. Conclusions

When Si substrate was processed by irradiation of infrared femtosecond laser pulses on the back surface, periodic structures similar to LSFL were formed. The periodic structure formation, morphology, and periodicity were studied by varying the laser energy and repetition rate. Also, irradiations were carried out on the front surface in order to compare with the periodic structures formed on the back surface. LIPSS was formed on the front and the back surface of Si substrate even by changing the direction of laser polarization. The LIPSS on the front surface were formed in perpendicular direction to the polarization plane of the laser while the periodic structures on the back surface were always formed parallel to the polarization. The period Λ of the back side LIPSS was smaller than the front side probably due to the decrease of light wavelength in bulk Si. When the back surface was in contact with water, the HSFL and LSFL were formed, depending on the laser conditions, with Λ smaller than that in air.

Our results demand further studies on the LIPSS formation, especially on the back surface and under liquids.

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