Sub-surface modifications in silicon with ultra-short pulsed lasers above 2 µm: role of the wavelength

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Abstract—The non-linear optical phenomena in silicon such as self-focusing and multi-photon absorption are strongly dependent on the wavelength, energy and duration of the exciting pulse, thus, resulting in a pronounced wave-length dependence of the sub-surface modifications with ultra-short pulsed lasers, especially above 2 microns. This wave-length behaviour is investigated for wavelengths ≥ 1950 nm at different pulse durations and energies both numerically and experimentally, and compared to already existing literature data at 1550 nm and 1030 nm.

Index Terms—Laser, mid-infrared, near-infrared, optical pulse generation, laser mode locking, laser tuning, laser material processing, silicon processing.

I. INTRODUCTION AND STATE-OF-THE-ART

Silicon wafers are currently largely separated from the bulk mono-crystalline Si-block using thin diamond saws, which introduces a high loss of the material (up to 50%) [1]. For extremely thin wafers with a thickness ≤ 100 µm the loss increases to ≈ 70% [1]. Thus, alternative methods for wafer separation are in development, such as epitaxial Si lift-off, stress-induced spalling, and smart-cut [2]. The latter technique employs the fact that by the introduction of defects in a target layer below the Si wafer this layer will be weakened, and thus the wafer itself can be removed. There are multiple techniques for introducing such defects in bulk silicon (Si). On one hand, the expansion coefficient difference for two materials can be used [3], without adding modifications inside the crystal. On the other hand, the crystal itself can be modified on the inside, for example by the use of a layer of porous Si [4], the use of high-energy protons [2] or the use of multi-photon absorption of laser pulses [5].

For the latter method several different approaches both in pulse energy, pulse duration and wavelength have been conducted earlier. The results are listed in tab:FormerExpData. Here it can be seen that even though a lot of work has been conducted at the common wavelengths such as 1064 nm and 1550 nm, way less work has been done at longer wavelengths. Furthermore the onlyienest work done at wavelengths longer than 1550 nm showed modifications below a layer of SiO2, but not within bulk Si.

The present work work focuses on investigation of the influence of the wavelength, pulse energy and pulse duration using both, numerical simulations and experiments for wavelengths between 1950 – 2400 nm, and compares with existing data for wavelengths between 1030 and 1550 nm. On the experimental side several different lasers have been used to modify a Si-crystal both on the surface and within the bulk material by the use of wavelengths ≥ 1950 nm. In order to optimize the process both in quality and speed, several parameters such as pulse energy, pulse duration and wavelength have to be optimized. It already has been shown that shorter pulses provide better results (in terms of cut quality and precision) compared to longer pulses [15, 16], while lowering the ablation threshold. Furthermore it has been shown that longer wavelengths in the mid-infrared range, i.e. longer than 2 µm, are beneficial for processing of bulk Si, due to the combination of lower non-linear absorption and higher self-focusing [17, 18, 19]. Both parameters can be combined in a so-called Figure of Merit (FoM)-value[19, 20], using eq. (1).

\[
FOM = \sum_k \frac{n_2(\lambda)}{\lambda \beta(K)} K^{-1}
\]

An optimal wavelength range can be found both based on eq:FOM and with simulations to be in the range of 2000 – 2300 nm [17]. The exact wavelength is depending on the exact value of the involved parameters, after for example the value for the non-linear refractive index \(n_2(\lambda)\) has measurement errors up to 30% [18, 19].

Several investigations already have been presented at lower wavelengths, both for surface ablation and in-depth modification of Si [21, 22, 23]. Furthermore different pulse
energies and -durations were investigated both in simulation and experiments [24, 25, 17, 26, 27], but until now to the best of our knowledge no investigation was done for pulses at wavelengths longer than 1550 nm, except in [24], where only the pulse propagation itself was considered, but not the temperature response of the material, and thus changing properties of the material.

Thus a combination of simulations and experiments was employed for wavelengths \geq 1550 nm, such that the correlation of the used values and model could directly be compared. In this paper the results of both simulations and experiments using lasers with a wavelength of 1965 nm, 2100 nm and 2350 nm at different pulse energies and pulse durations \leq 2 \text{ps} are shown. In order to simulate the propagation of the pulse through the material, the non-linear Schrödinger equation is used [24]:

\[ \partial_z E = \frac{i}{2k_0} \nabla^2 E + \frac{i k_0 n_2}{\hbar_0} |E|^2 E - \frac{1}{2} \sum K \beta^{(K)} |E|^{2K-2} E - \frac{\sigma}{2} (1 + i \omega_0 \tau_c) NE \]  

(2)

It describes the propagation of a laser pulse through bulk Si, while taking the non-linear refractive index \( n_2 \), based on [18, 19]) and thus the self-focusing of the pulse, the non-linear multi-photon absorption for \( K \)-photon-absorption \( \beta^{(K)} \), the absorption and the diffraction due to generated free carriers into account. In this case, only two- and three-photon absorption was taken into account, after the investigated wavelengths were in a range where the single-photon-absorption and higher-photon-absorption could be neglected [28]. It can be seen that for higher initial intensities the importance of the non-linear effects increases (as for example seen in Fig. 1).

An additional effect which has to be taken into account is the paraxial approximation. It introduces several simplifications into the beam propagation equation, but is only valid until a certain beam diameter [29]. Thus, if the beam is focused too strongly (due to the optical Kerr-effect), eq. (2) which uses that approximation loses its accuracy, returning in wrong results. This problem will be investigated in sec:NumSim.

There are different approaches for calculating the response of the material if irradiated with ultra-short pulses, which are extensively discussed in [30], such as the two-temperature-model (TTM) (and in extension the three-temperature model [31]), a hydrodynamic approach or a molecular dynamics-simulation (MD). Furthermore, hybrid models such as a combination of a TTM and a MD are suggested. To simplify the calculations, only the generated carrier density will be taken into account for this paper. This value can be calculated by using [24]

\[ \partial_t N = \sum K \beta^{(K)} |E|^{2K} \]  

(3)

However, for future investigations a more extensive model is necessary.

II. NUMERICAL SIMULATIONS

An initial fast comparison of pulses at different wavelengths can be done using eq:NonlinearSchrödinger, by comparing the transported energy towards the focal spot. A result of those calculations for the wavelengths 1550 nm, 2100 nm and 2400 nm at the pulse energies 5 nJ and 1 \mu J can be seen in figure 1. There, while the lower-energy pulse can deposit more energy at the focus point at 1700 nm than at the longer wavelengths, it is the other way around for the higher-energy pulses, and the longer wavelengths can transport more energy towards the focus point, where the energy is absorbed. The reason for this are the non-linear properties in Si, which are changing depending on intensity and wavelength. Thus the absorption for the shorter wavelengths is higher already before the focus point, leading to early absorption and heating of the material at non-targeted positions.

![Figure 1: Energy of a pulse with different wavelengths and energies, depending on the position within the material. The focal length of the used lens is f=8 mm, with the focal point at f=4 mm inside the material. The pulse duration is \( \tau = 500 \text{fs} \).](image)

When considering the achieved intensities, one can see in Fig. 2a both the influence of the nonlinear refractive index, the absorption of the pulse and the collapse of the pulse.

![Figure 1: Intensity and absorption of a pulse with different wavelengths, depending on the position within the material. The focal length of the used lens is f=8 mm, with the focal point at f=4 mm inside the material. The pulse duration is \( \tau = 500 \text{fs} \).](image)

In this figure the self-focusing of 2100 nm latter wavelength starts significantly earlier than for the other two wavelengths. This leads to a modification zone which is spread out much more. Higher intensity also means higher absorption, which can be calculated using [24]

\[ \partial_t W = \sum_K \beta^{(K)} |E|^{2K} + \sigma N |E|^2 \]  

(4)

and is shown in Fig 2b. As one can see, the absorption does not only depend on the non-linear absorption, but also on the free carrier density, which in turn is increased by the pulse itself.
(compare eq. (3)). This generated free-carrier density is shown in Fig. 3.

Again, the pulse at 2100 nm can generate more free carriers, compared to the other two wavelengths, which in turn increases absorption and diffraction again. When considering extended models, such as Two-Temperature-Models (as for example in [23, 26]), the free carrier density also plays an important role by coupling the energy of the generated free carriers to the lattice itself by the coupling constant [23]:

$$\gamma(N) = \frac{3k_B N}{\tau_0 \left( 1 + \left( \frac{N}{N_{cm}} \right)^2 \right)}$$

(5)

Thus, a higher free carrier density makes the energy transfer from the excited carriers to the lattice more efficient. This assumption still has to be investigated further in future research by coupling the pulse propagation to a two-temperature model. Finally, two different processes can lead to melting[32, 33]. On the one hand, the carrier density can be increased up to a critical carrier density[26, 34]. At that density enough free carriers are excited that the material can show a behaviour called "non-thermal melting"[35, 36, 33, 16]. This density (\(n_{c,m} \approx 10 \times 10^{22} \text{cm}^{-3} = 10 \times 10^{18} \text{m}^{-3}\) [34]) is reached when approximately 10 – 20% of the valence electrons are excited. This state shows a behaviour similar to melting, while the lattice itself is still below melting temperature. The other process is conventional thermal melting, when the lattice reaches a temperature above the melting temperature. This temperature increase comes from the energy transfer from the carriers, the higher the electron temperature, the higher also the resulting lattice temperature. Based on the numerical simulation data shown above (Fig. 2,3) and the experimental data shown below in Section III it can be concluded that the created modifications in Si have a thermal nature, the generated free-carrier density did not exceed the critical density. Therefore, the initial assumption, that longer wavelengths in the range of 2000 – 2200 nm are more efficient for modifying monocrystalline Si, can be confirmed by our numerical simulations. Still, this has to be compared to experimental results, too, which will be done in Section III. In addition, further, more extensive numerical analysis has to be done in the future, as mentioned above.

III. EXPERIMENT & DISCUSSION

3.1 Laser sources
In order to get experimental data which can be used as verification to the numerical results, modifications both on the surface and below the surface of Si-wafers were made using three different lasers: A fiber-MOPA operating at \(\lambda = 1965 \text{ nm}\), \(\tau_p = 2 \text{ ps}\) and \(W_{\text{max}} = 1150 \text{ nJ}\) (see subsection 3.1.1), a solid-state laser operating at \(\lambda = 2090 \text{ nm}\), \(\tau_p = 0.5 \text{ ps}\) and \(W_{\text{max}} = 1150 \mu\text{J}\) (see subsection 3.1.2) and a solid-state laser operating at \(\lambda = 2350 \text{ nm}\), \(\tau_p = 1.1 \text{ ps}\) and \(W_{\text{max}} = 20.3 \text{ nJ}\) (see subsection:SSL2350).

3.1.1 2-µm Fiber MOPA
The fiber-MOPA (labeled laser "A" in this paper) consists out of an oscillator, mode-locked by a SESAM, and two amplification stages. Here, the oscillator generates laser pulses at a wavelength of \(\lambda = 1965 \text{ nm}\) with a repetition rate of \(f = 7.6 \text{ MHz}\), a pulse duration of \(\tau = 2 \text{ ps}\) and a pulse energy of \(W = 0.1 \text{ nJ}\). By using a pre-amplifier this pulse energy is amplified to \(W = 0.5 \text{ nJ}\). Finally, the pulse is amplified in a second amplifier to a pulse energy of \(W_{\text{max}} = 0.98 \mu\text{J}\).

3.1.2 Solid state laser at \(\lambda = 2090 \text{ nm}\)
For experiments at 2090 nm we used a compact ultrashort pulsed fiber laser based Ho:YAG amplifier system from ATLA Lasers AS that has been developed following [37] (labeled laser "B" in this paper). The latter two-stage system first converts femtosecond pulses at 1045 nm to 2090 nm using an optical parametric amplifier, generating pulses of about 0.5 ps duration. After stretching and amplitude premodulation the seed pulses are further amplified in a Ho:YAG solid-state amplifier and recompressed. At 10 kHz repetition rate the system delivered up to 30 µJ pulses from the seed stage with 460 fs duration and up to 3 mJ pulses with \(\approx 3.5 \text{ ps}\) duration from the amplifier stage.

3.1.3 Solid state laser at \(\lambda = 2350 \text{ nm}\)
The solid-state laser (labeled laser "C" in this paper) was a mode-locked Cr:ZnS oscillator from ATLA Lasers AS working in the normal dispersion regime [38]. The standard 70 MHz delta-cavity was expanded by the Herriott-type multi-pass cavity (see the cavity schematics on fig:fig1) which allowed achieving pulse repetition rate of 8.62 MHz.

![Figure 4: Extended cavity Cr:ZnS chirped-pulse oscillator setup (CPO).](image-url)
The combination of dispersion-managed mirrors and material dispersion compensation in the cavity ensured the normal-dispersion operation regime of the laser. The laser generated linearly-chirped pulses centered at 2350 nm with duration around $\tau=1.7$ ps and pulse energy up to 48 nJ (see fig:fig2). Around 50% pulse energy was lost in the isolator, beam delivery optics and focusing objective, resulting in slightly above 20 nJ delivered to a surface of silicon sample. The diffraction-limited focal spot width at the surface (single aspheric lens) was 1.6 $\mu$m.

In this comparison in Fig. 6 it can be seen that the cut using the longer wavelength is significantly smaller in diameter (1 $\mu$m compared to 2 $\mu$m) and cleaner (i.e. less residual around the cut), thus suggesting that using a longer wavelength might be beneficial for cutting of Si. Nevertheless this reduction can also originate from either a smaller spot size (as described in subsections 3.1.1 and 3.1.3).

Assumed the beam was focussed perfectly onto the surface, the focal spot size for laser A is 4.25 $\mu$m, while for laser C the spot size is 1.6 $\mu$m. Furthermore, it is assumed that the absorption for laser A was purely based on two-photon-absorption, and the absorption from laser C was purely based on three-photon-absorption. That means that the effective spot size (if the threshold is reached for the whole Gaussian pulse) is $\approx 3$ $\mu$m for laser A, and 0.92 $\mu$m for laser C. While the result for laser C is close to the measured width of the lines, the calculated effective spot width for laser A is significantly larger than the measured line width. This can be related to several things, such as an incorrectly measured beam width (the effective spot size can be reached for a beam width of $\approx 1.6$ mm), or a higher modification threshold, thereby also reducing the effective spot size. Nevertheless, both possibilities have to be investigated in the future, after none can be excluded based on existing data at the moment.

Furthermore the lasers were not only focused on the surface, but also below the surface for making modifications there. Those modifications were observed both using a transmission FTIR and an infrared microscope. The FTIR microscopy was carried out in transmission mode on a Bruker Hyperion 3000 microscope with a 64 x 64 pixel mercury-doped CdTe focal plane array detector interfaced to a Bruker Vertex 70v FTIR spectrometer. A 10X Cassegrain objective was used for imaging. Background spectral images were recorded on an empty sample stage, and used to calculate extinction spectra at each pixel after measuring the laser processed Si sample. For spectral acquisition, 100 scans with a spectral resolution of 4 cm$^{-1}$ were averaged. Spectral images were obtained from two spectral regions. Extinction maps from the region 3.965 – 3.027 $\mu$m (2522 – 3304 cm$^{-1}$) were obtained as false-coloured maps from integrating extinction without baseline correction. As the system does not show any absorption in this spectral range, extinction here is caused by scattering. Maps were also produced in the spectral region 10.320 – 7.553 $\mu$m (969 – 1324 cm$^{-1}$), containing the absorption peaks of Si-O stretching modes. In this region, a linear baseline was subtracted from the spectra between the left and the right end of the spectral range. In that manner, maps show the distribution of absorption from Si-O modes. Results of those measurements both for the laser operating at 1965 nm (laser A), for the laser operating at 2090 nm (laser B) and for the laser operating at 2350 nm (laser C) are displayed in Fig. 7 and 8.

![Figure 5: Input-output characteristics (a), optical spectrum (b), and interferometric autocorrelation trace (c) of the extended cavity Cr:ZnS CPO.](Image)

3.2. Experimental setup

All lasers were coupled to a processing stage. The light was focused using a lens with varying focii, typically $f=4$ mm either onto the surface of the sample (as in fig:SurfaceCut) or inside the sample (as in fig:vertlines1, for example). The sample itself was mounted onto a four-axis stage, with two of them controlled by an electronic controller. This allowed precise movement with a fixed speed, variable between several $\mu$m s$^{-1}$ and several mm s$^{-1}$.

3.3 Investigation of the processed samples

After processing the samples were investigated using three different techniques, optical microscopy, transmittive IR microscopy and SEM imaging. The first two techniques are non-destructive, while the imaging using the SEM required the breaking of the sample to investigate the cross-section of the sample. In addition the optical microscopy only could be applied on the surface, while the transmissive IR microscopy could be used to look through the sample and investigate subsurface modifications without damaging the sample.

The optical microscope was used to investigate the lines created on the sample, and to get an initial comparison between the wavelengths. The used lasers were the fiber-MOPA, operating at $f = 7.6$ MHz and $W = 28$ nJ, and the second solid-state laser, operating at $\lambda = 2350$ nm, $f = 3.7$ MHz and $W = 17$ nJ.

![Figure 6: Line on surface, created with $\lambda=1965$ nm and $\lambda=2350$ nm](Image)
Figure 7: Transmission IR images of a line below the surface of Si. Created with laser A operating at $\lambda$=1965 nm and $W$=180 nJ. Figure a) shows the mapped integrated extinction in the range 3.965 – 3.027 $\mu$m and b) the mapped integrated absorbance in the region 10.320 – 7.553 $\mu$m. The scale bar applies to both a) and b). c) shows the example spectra at the spots marked in the respective colour in a). As in fig:20180314line-5, outside the IR maps the continuation of the sample is shown as a visible image.

Figure 8: Transmission IR image of a line created by focusing laser C with $\lambda$=2350 nm below the surface of silicon. Right: False colored absorbance scale in the wavelength range 3.965 – 3.027 $\mu$m. The visible part of the image left and right of the central false coloured image presents a continuation in the visible of the area imaged in the IR. There no modification can be seen, thus indicating that the modification resulting in increased absorption only exists below the surface.

Furthermore, a transmissive IR-microscope operating at $\lambda$ = 1300 nm was designed. It also could be used as a non-destructive detection method of sub-surface modifications in bulk Si. Here, to make the modifications more visible in the microscope, the sample was moved parallel to the beam, while the samples shown in Fig. 7 and 8 were moved perpendicular to the laser beam. This movement resulted in visible spots at the surface of the sample, shown in Fig. 9. The lines were created using laser B, at a wavelength of 2090 nm.

The sample was moved vertically with a speed between 0.2 – 20 $\mu$m s$^{-1}$ from the bottom of the sample towards the top surface, while the pulse energy was varied between 3.7 and 7.7 $\mu$J. A side view of a part of the sample can be seen in fig:vertlines1.

As a next step, another sample was modified using the same parameters, but moved perpendicular to the beam. This sample is shown in fig:Sample18.

Figure 9: Vertical lines created in monocrystalline Si, viewed using a transmission IR microscope from the top of the sample. Pulse energy for the top row was 3.7 $\mu$J, while the pulse energy for the bottom row was set to 7.7 $\mu$J. The movement speed of the stage decreases from left to right.

Figure 10: Vertical lines created in monocrystalline Si, viewed using a transmission IR microscope from the side of the sample. The influence of the movement speed and the pulse duration can be seen, even though the view partially is obstructed due to several rows lying behind each other.

As a next step, another sample was modified using the same parameters, but moved perpendicular to the beam. This sample is shown in fig:Sample18.

Figure 11: View of the surface of the sample, using the optical microscope.

Figure 12: Horizontal lines created in monocrystalline Si, watched from the side. Line stacking clearly is visible.

It can clearly be seen that there are modifications below the surface between line M4 and M6, even though they are not continuous. It also has to be noted that line M4, M5 and M6 are
3.4 Discussion

Three different lasers operating at central wavelengths of 1965 – 2350 nm have been presented. All lasers were used for making sub-surface modifications of bulk Si, while varying the pulse energy and pulse duration. Afterwards all samples used in modification experiments were investigated using both non-destructive and destructive techniques. It could be shown that all three lasers could produce sub-surface modifications.

The modifications were investigated more thoroughly for \( \lambda = 2090 \text{ nm} \). There it could be shown that the modifications are both visible using a classical transmissive IR microscope and SEM. Both methods confirmed the measurements from each other. As already mentioned in sub-section 3.3, the modifications are not perfectly shaped around the focal spot, but rather elongated and start significantly before the focal spot. One possible explanation for that is due to nonlinear absorption happening already earlier, i.e. the critical intensity for absorption is reached already before the focal spot, starting the modification process earlier. A similar behaviour can be seen in Fig. 2a.. One would expect the intensity to rise to one single peak, but instead it hits a maximum value, before staying at that level for several and decreasing again afterwards. In that period the pulse energy is absorbed within the material, and depending on the pulse energy is leading to modifications at points which are not targeted.

IV. Summary & Outlook

In conclusion we have shown both in experiments and numerically that 1) there is an optimal wavelength around 2.1 micrometers, where modification of the silicon inside the crystal happens at lower pulse energies at the same other experimental parameters, and 2) by using wavelengths \( \geq 2 \text{ \mu m} \) modifications within bulk silicon can be done already at pulse energies lower than the energies reported in the literature, such as in [13], going down to nJ pulse energies. In addition it could be shown that while no visible damage on the surface was observed, IR images show a contrast in the volume of the material, a benefit compared to for example [11], where the authors experienced difficulties producing modifications inside the silicon volume without damaging the surface. This knowledge can be used not only for creating a weakened layer below the surface, but also for example for creating wave guides. When comparing to the pulse values given in [6, 9, 8, 11, 12, 14], it also can be seen that the generated sub-surface modifications were created at comparable or lower pulse energies. This also indicates that modifications at longer wavelengths \( \geq 1550 \text{ nm} \) have a lower threshold than at shorter wavelengths, as predicted in Section II.

Furthermore due to the non-linear behaviour of multi-photon absorption, structures can be created with a resolution below the wavelength, something which is not possible with single-photon absorption. This was demonstrated in Fig. 6. One reason for that is the nonlinear absorption is starting already before the focal spot, thus creating modifications with sizes larger than the pulse wavelength (as it can be seen in Fig. 12 and 13).

This effect could be managed using lenses with higher NA, and thus a larger focusing angle. That would reduce the intensity before the focal spot, ideally as close to the focal spot as possible, before nonlinear absorption and -focusing starts and creates modifications. Even though it could be shown that the used lasers were capable of creating sub-surface modifications, this behaviour prevents using them in the current configuration for the purpose of creating defects below a silicon wafer. Targeting a wafer thickness of \( \leq 100 \text{ \mu m} \) it is not beneficial to destroy \( 300 - 400 \text{ \mu m} \) of the silicon below for creating a weak layer, allowing the top layer to be removed. This would still lead to losses of \( 75 - 80 \% \), which is no improvement compared to currently employed techniques [1].

Finally the exact nature of the modified Si on the physical-chemical level has to be investigated in the future in more detail in order to gain a better insight, and to improve the process. This can be done both experimentally (by, for example, using micro-Raman analysis of the sample cross section) and by numerical simulations. For the latter approach more advanced models will be used, such as a two-temperature-model (TTM), as described in [23, 26], a three-temperature model [31] or a model combining several different approaches, such as described in [15].

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