Laser-Ablated Silicon in the Frequency Range From 0.1 to 4.7 THz

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***Abstract*—The optical performance of high-resistivity silicon with a laser-ablated surface was studied in the transmission mode in the frequency range of 0.1–4.7 THz. A reciprocal relationship between the transmission brightness and the surface roughness was observed at discrete THz frequencies. The measured dispersion was reproduced by the THz wave scattering theory using an effective refractive index model. No significant differences between the samples processed either with ps- or ns-duration laser pulses in ambient air or in argon enriched atmosphere were found in the THz regime. It was demonstrated that the majority of optical losses of the silicon with the laser modified surface were due to the scattering of THz waves and not due to the absorption in silicon-compounds formed during the laser ablation.**

***Index Terms*—Diffraction, laser-ablation, scattering, terahertz (THz) waves.**

I. Introduction

ELECTROMAGNETIC waves of terahertz (THz) frequency gain more attention from scientists and engineers due to new emerging applications of THz imaging in astronomy, medicine, art conservation, security, materials inspection, etc. [1]–[5]. Off-axis parabolic mirrors and refractive lenses are available commercially and very often are employed in THz imaging setups. Compact diffractive lenses such as multi-level phase Fresnel lenses (MPFLs) and Fibonacci lenses have been developed on high-resistivity silicon (HR-Si) in order to reduce the complexity of the THz systems [6]–[9]. The advantages of diffractive optics have been experimentally confirmed by demonstrating almost diffraction-limited imaging results [10], [11]. Silicon MPFLs have been fabricated using photo-lithography and the reactive ion etching technique [7] or using the maskless direct laser ablation (DLA) technology [12]. Moreover, the maskless laser patterning has been used to introduce additional functionalities in the MPFL, such as the phase-alteration structures working as both focusing and anti-reflective surfaces [13].

The DLA technology makes the THz components fabrication process flexible and simple to redesign [12]. Nevertheless, it is still essential to reduce the production time in order to ensure affordable final product cost. The material removal rate in the DLA process depends on the irradiation parameters such as laser fluence at the center of the focused laser spot F_0 and laser spot overlap. At the same time, the ablation quality, i.e., roughness of the laser-ablated surface, also depends on these parameters. It was shown that for a fixed pulse repetition rate the efficient ablation regime (maximal removal rate per average laser power) is achieved when the fluence is approximately 7.4 times larger than the threshold ablation fluence [14].

In many cases, this efficient regime also leads to high fabrication quality with a minimum surface roughness of the ablated surface [15]. So, in theory, to achieve the maximal ablation efficiency and best surface quality, the spot size of the laser source should be adjusted to the fluence to meet the most efficient ablation regime. However, in microstructuring applications in the efficient ablation regime, the spot size would be too large to obtain fine structural features. For example, fabrication of high-frequency MPFLs may require a spot size of 20 μm in diameter or even smaller [6], [9]. Therefore, it may be beneficial to exceed the optimal fluence. When the fluence is raised beyond the optimal value, the ablation rate increases; but the laser power is used inefficiently. Also, as the material is removed faster, the fabrication is not so precise, and the surface roughness tends to increase [15]–[17]. High surface roughness can lead to the scattering of THz radiation (especially in the high-frequency part of the THz spectrum). Furthermore, even using low fluence for the fabrication, the composition of the top layer of the silicon wafer may be altered due to the formation of silicon oxide [18] or amorphous silicon [19].

In this article, the performance of HR-Si with the laser-ablated surface was studied in the transmission mode in the frequency range from 0.1 to 4.7 THz. The influence of the fabrication parameters and processing environment on the THz performance of the laser-ablated silicon wafer was investigated. The aim of this article was to find how rapid silicon processing with high laser fluence can impact the optical properties of THz components in the relevant spectral range. In particular, laser

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ablation with picosecond and nanosecond pulses was performed. In total, 37 samples on HR-Si were fabricated, changing the processing parameters such as the pulse repetition rate, pulse energy, impulse overlap, and processing environment.

II. Samples and Experimental Setup

The samples were fabricated using a galvanometric scanner (HurryScan 14 from Scanlab), a 160-mm focal length *f*-theta lens (LINOS/Rodenstock), and a fundamental harmonic of the laser: Atlantic-60 (max. rep. rate 1 MHz, max. pulse energy 60 μJ, 13 ps, 1064 nm, Ekspla Ltd.) and Baltic-HP (max. rep. rate 100 kHz, max. pulse energy 120 μJ, 10 ns 1064 nm, Ekspla Ltd). For all experiments, a fixed repetition rate, which could be provided by both lasers (100 kHz), and an 18-μm diameter spot size of the focused laser beam were used. The majority of the samples were fabricated in ambient air. However, a few of them were processed in an argon (Ar) atmosphere at a pressure of 1 or 2 atm. The samples were fabricated from a 500 $\pm$ 25 μm thick, both-sides polished, (100) crystal orientation high-resistivity float zone silicon wafer that was similar to the ones used elsewhere [9], [12].

III. Results and Discussion

The silicon was laser-ablated into a depth of about 30 $\pm$ 4 μm in order to develop a rough surface with an area of 6 $\times$ 6 mm^2. When a surface roughness (R_a) higher than 2000 nm was required, the deeper layers were ablated using nanosecond duration laser pulses. Fabrication regimes allowing the production of such rough surfaces provided material removal thickness ranging from 4 to 40 μm in a single scan. Therefore, in high roughness samples, the depth was not preserved to be constant.

The THz performance of the samples was investigated in the transmission mode in the frequency range from 0.1 to 4.7 THz using THz imaging and THz time-domain spectroscopy (THz TDS) systems described elsewhere [6], [9], [11]. A stylus profiler (Veeco Dektak 150) and a scanning electron microscope (SEM) (JEOL JSM) were used to characterize the morphology of the samples. An elemental analysis was performed using an energy dispersive spectroscopy (EDS) attachment (Oxford instruments X-Max).

Representative surfaces of the HR-Si after ablation with ps duration laser pulses are shown in Fig. 1. The measured dependence of the removal rate and the surface roughness on the fluence are shown in Fig. 2. With the constant spot size value of 18 μm, the optimum fluence value (maximal removal rate per average power) $F_{0\mathrm{opt}}$ was found to be about 3.4 J/cm^2 [see Fig. 2(b)] and was in good agreement with the theoretical ablation rate model [20]–[22]. The smallest surface roughness was detected at the optimal and slightly higher fluence values [see Fig. 2(c)]. At $F_{0\mathrm{opt}}$, the removal rate up to 60 μm^3/min was achieved. However, as can be seen from Fig. 2(a) and (c), the material removal rate can be further increased by several times with the increase of the fluence, if a moderate increase in surface roughness and not optimal utilization of the laser power are acceptable. The SEM images and surface profiles of the selected samples are shown in Fig. 1. They demonstrate that

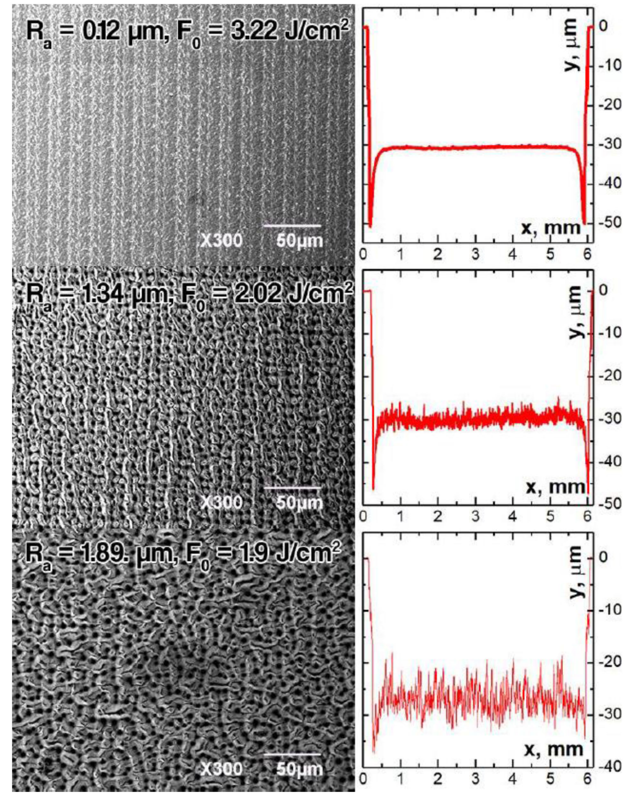

Fig. 1. SEM image (left) and depth profile (right) of the silicon surface, processed with 13-ps duration 100-kHz rep-rate laser pulses and an 18-μm focused beam spot-size with 60% spot overlap. The measured surface roughness (R_a) and the used laser fluence are indicated for each case.

$F_{0\mathrm{opt}}$ can be used to ablate the silicon surface with minimal corrugations in the surface morphology, however, even if the fluence is not optimal and the surface roughness increases, the corrugations may be of much smaller scale than the wavelength of THz radiation of interest.

The X-ray EDS analysis of the oxygen and silicon atomic percentage ratio was performed for the HR-Si laser-ablated in ambient air and Ar enriched atmosphere. The results are shown in Fig. 3. It is of interest that the relative amount of oxygen depends on the surface roughness rather than the research atmosphere (air or Ar). This can be explained by the fact that the geometric area of a rough surface is larger than that of a smooth surface and therefore an oxidation results in a larger scale [23]. Also, relatively high oxygen content on the surface may be caused by oxygen contamination from air. The ratio between oxygen and silicon was found independent on whether the ablation was performed in ambient air or Ar atmosphere.

In order to increase the roughness of the laser-processed silicon surface, another set of samples was fabricated using DLA with ns-duration laser pulses. The surface morphology images of the selected samples are shown in Fig. 4. The samples demonstrated R_a values from 1 up to 60 μm achieved by varying the laser beam fluence and spot overlap.

The transmittance of the samples with various surface roughness was measured by using the THz TDS system in the frequency range of 0.1–3 THz. The results are shown in Fig. 5(a). In

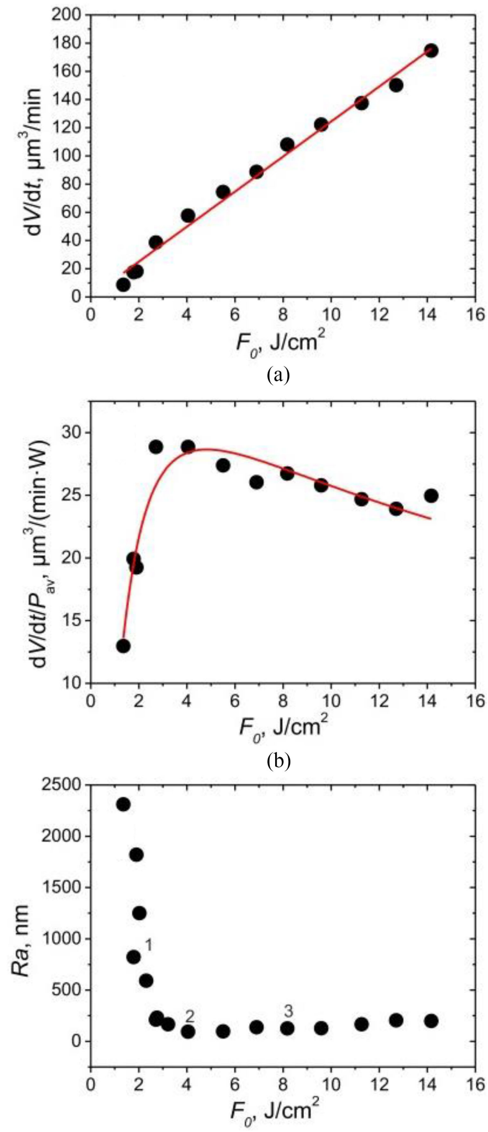

Fig. 2. Dependence of the ablation rate and the surface roughness on the laser fluence using 13-ps duration laser pulses. (a) Removal rate as a function of the fluence. (b) Removal rate per average power as a function of the fluence. (c) Surface roughness as a function of the fluence. The laser fluence was measured in the centre of the laser spot.

all spectra, the transmittance was almost constant up to the critical point after which it started to decrease. The critical frequency depended on the surface roughness value: it was lower when Ra was higher. Laser-ablation of silicon using various regimes did not alter the THz transmission if small R_a values were preserved. More specifically, the laser-ablated silicon surface did not contribute to the absorption up to 3 THz if $R_a = 1.7\,\mu$m or smaller. This is an important result for the application of the DLA technology in components fabrication for the THz frequency range. Moreover, we experimentally confirmed that deterioration of the THz performance of the laser-ablated component is due to the design and its implementation issues, rather than due to the laser-ablated surface absorption losses, as it has been previously assumed [6].

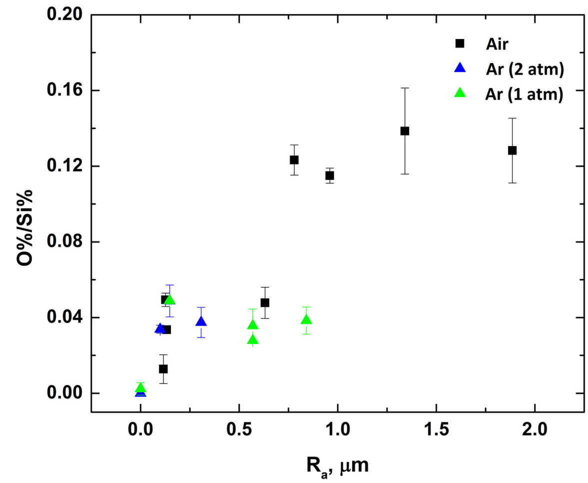

Fig. 3. Dependence of the oxygen and silicon atomic percentage ratio on the surface roughness of the laser-ablated HR-Si.

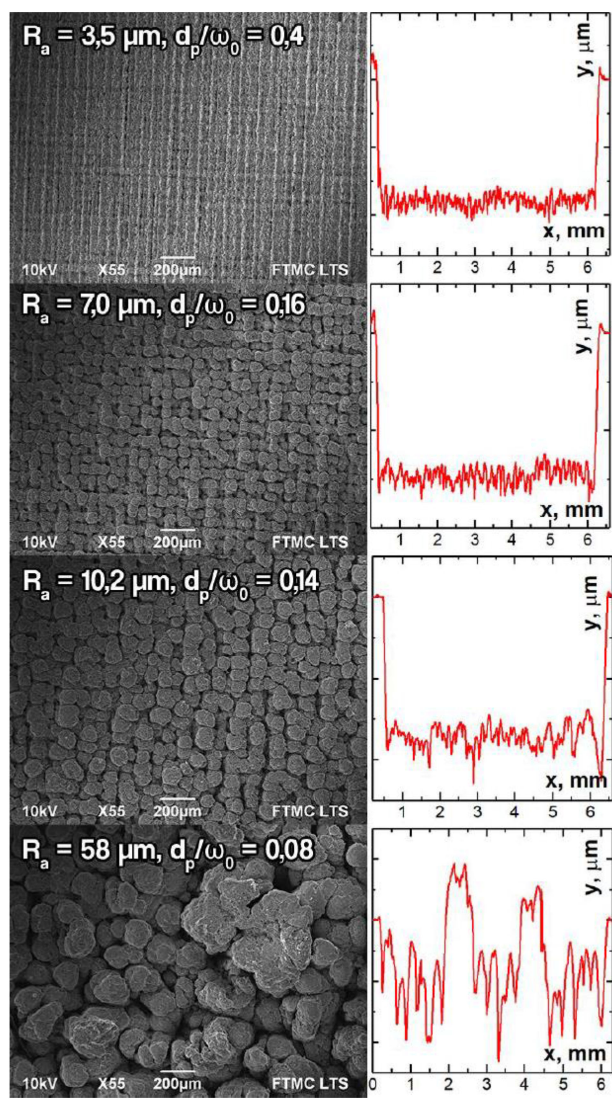

Fig. 4. SEM images (left) and depth profiles (right) of the silicon surface processed with 10-ns duration 100-kHz rep-rate laser pulses. Samples were fabricated using different value of the laser beam fluence and the spot overlap (d_p). The measured surface roughness (R_a) is indicated for each case.

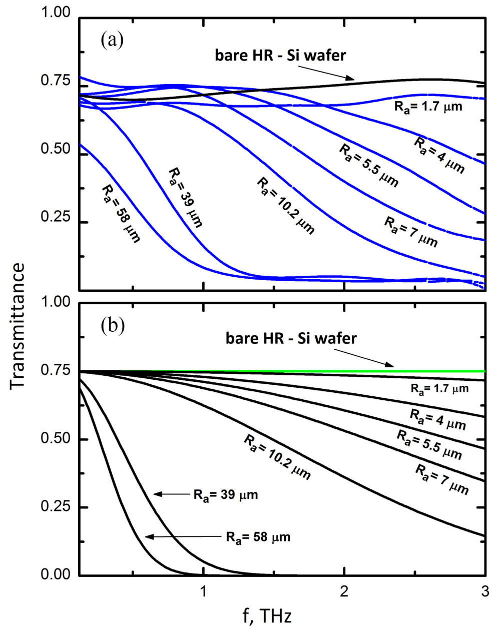

Fig. 5. Transmittance spectra of laser-ablated silicon samples (a) measured and (b) calculated using (2). R_a indicates the surface roughness.

Scattering losses from the rough surface at normal irradiation incidence angle can be expressed as [24]

$$R = R_0 \exp\left[-\left(\frac{4\pi\sigma}{\lambda}\right)^2\right] + R_0 \frac{2^5\pi^4}{b^2}\left(\frac{\sigma}{\lambda}\right)^4 (\Delta\theta)^2 \qquad (1)$$

where R_0 stands for the reflection from the smooth silicon surface, σ is the root mean square value of the surface roughness, λ is the irradiation wavelength, b is the average surface slope, and $\Delta\theta$ is the view angle of the measurements. In the case of $\sigma << \lambda$, the second term can be omitted, and scattering losses will solely depend on the surface roughness

$$R = R_0 \exp\left[-\left(\frac{4\pi\sigma}{\lambda}\right)^2\right]. \qquad (2)$$

The transmittance dispersion occurring due to the surface roughness of laser-ablated HR-Si was calculated using (2) with assumption $\sigma \sim R_a$. The results are shown in Fig. 5(b). Comparing the experimental and theoretical data it was confirmed that the transmittance drop at the specific surface roughness values was caused by the scattering of THz waves from the ablated silicon surface when the irradiation wavelength and surface corrugation size become comparable.

We also investigated scattering losses at higher frequencies measuring the THz transmittance of all samples at discrete frequencies provided by a THz quantum cascade laser. The results are shown in Fig. 6. The difference between the THz transmittance values of the samples produced in ambient air and those produced in Ar-enriched atmosphere were not obvious and conclusive. The spread of the measured data was in the range of about 15%–25%. The THz transmittance values of different samples, fabricated in the ps-regime, were averaged. The results are summarized in Table I. At 4.7 THz and 2.5 THz, the laser

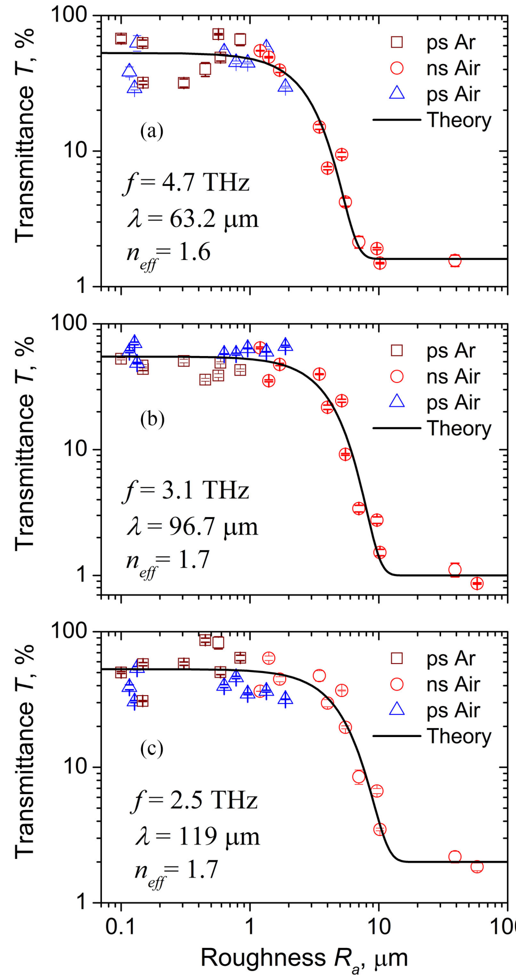

Fig. 6. Dependence of the transmittance on the surface roughness (R_a) for discrete frequencies. (a) 4.7 THz. (b) 3.1 THz. (c) 2.5 THz. The solid line was calculated using (2). As indicated, the samples were processed by the DLA technology in the ns- or ps-regime in ambient air or inert Ar gas.

TABLE I
AVERAGE TRANSMITTANCE OF LASER ABLATED SILICON SAMPLES AT DISCRETE THZ FREQUENCIES

Frequency, THz	Average transmittance (ambient air), %	Average transmittance (Ar gass), %
4.7	45.2	60.5
3.1	60.5	41.0
2.5	39.0	66.2

ablation of HR-Si in Ar tends to provide a positive effect on the transmittance values. However, at 3.1 THz frequency, the samples processed in Ar demonstrated, on average, a somewhat smaller transmittance. Taking into account every measured sample (see Fig. 6), the results distribution demonstrated no conclusive difference between the data shown in brown triangles (Ar) and blue squares (Air) points.

To fit the experimental results with the theory, the effective refractive index n_{eff} of the coarse, laser modified surface, which acts as a scattering media, has to be taken into account [13]. A product of n_{eff} and R_a was used in modeling instead of σ. Good agreement between the experimental and modeling results (see Fig. 6) allowed us to conclude that the surface of laser-patterned HR-Si had the effective refractive index of about 1.7.

IV. Conclusion

The optical performance of the HR-Si with the laser-ablated surface has been investigated in the transmission mode in the frequency range from 0.1 to 4.7 THz. The difference between the samples fabricated either with ps- or ns-pulse duration lasers in ambient air or Ar-enriched atmosphere was found to be small in the THz regime. Nevertheless, reciprocal relationship between the THz transmission and the surface roughness values has been observed. The measured dispersion at discrete frequencies was reproduced by the model of THz wave scattering on rough surfaces, demonstrating that the majority of optical losses are due to the scattering but not due to the absorption in laser-ablated silicon compounds, formed during the fabrication process. This is the first direct demonstration that the DLA technology is suitable to functionalize the silicon surface by adding properties of diffusion scattering and specular reflection without the introduction of absorption losses in the THz range. The results seem to be promising for the development of phantoms of skin and biological tissues.

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