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Periodic Surface Structure Creation by UV Femtosecond Pulses on Silicon

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Abstract. Laser-induced periodic surface structures are created on Si (100) and Si (111) wafers by 500 fs laser pulses at 248 nm. The periodic structure is concentric and highly regular. The spatial period is consistently varying between 1.1 μm and 3.3 μm in the radial direction. It is shown that the fluence of the irradiation at the same pulse number determines the size of the area where the periodic structure is created and for the same fluence the pulse number determines the regularity of the created grooves by melting processes. The origin of this structure is identified as the inhomogeneity of the laser beam profile caused by Fresnel diffraction close to the focal plane. Further improvement of the formation of periodic structure with femtosecond laser pulses is suggested.

INTRODUCTION

The use of laser pulses for material ablation is widely applied in material processing and in industry [1]. Laser ablation of material is a complex process involving several phases and multiple length and time scales [2,3,4]. Ultrashort laser pulses are especially promising for micromachining of various materials, since high energy density can be transferred to the materials in such a short timeframe, that thermal effects on the material are minimal. Femtosecond laser nano- and microstructuring has been investigated for the past decade [5-9]. The formation of laser-induced periodic surface structures (LIPSS) on silicon and on other materials is extensively investigated both theoretically and experimentally [10-17]. Irradiation of the surface with multiple linearly polarized femtosecond laser pulses can lead to the formation of periodic surface structures in two different ways; the high spatial frequency and the low spatial frequency LIPSS. In case of the high spatial frequency LIPSS the period of the ripples is significantly smaller than the wavelength and it appears after hundreds to thousands of laser pulses at fluence levels below the single-pulse ablation threshold. The origin of this sub-wavelength periodic structure is unclear and it forms the basis for several studies. [18-20] On the other hand low spatial frequency LIPSS are observed after a few laser pulses somewhat above the ablation threshold with a spatial period similar to the wavelength. In case of normal incidence the created periodic structure is always perpendicular to the polarization vector of the linearly polarized beam. This structure is formed due to interference of the incident laser beam with the surface-electromagnetic wave created by the irradiation. Several groups also reported that the excitation of surface plasmon polaritons in the generated quasi free electron plasma plays a crucial role during the early stage of the low spatial frequency LIPSS formation [14, 15, 20].

LIPSS formation on silicon and on other materials was investigated predominantly by IR fs pulses, however several paper reported LIPSS formed by UV fs pulses [21-23]. The created spatial periods were similar compared to

the IR case. Ordered structures with characteristic sizes from several μm down to 200 nm were observed. It is conspicuous that the irradiation dose for nano- and microstructure creation is lower for UV fs pulses.

In this paper we are reporting about laser-induced microstructures on two types of silicon (Si (100) and Si (111)) wafer by UV femtosecond pulses with a fabrication process which seems inherently different from the above mentioned types. We examine the dependence of the created structures on the irradiation dose. We identify the process of micromachining and suggest further improvement for this type of surface structure creation.

EXPERIMENTAL ARRANGEMENT

The working tool for the experiments was a so-called Szatmari-Schafer type dye/excimer hybrid UV short-pulse laser system [24, 25]. In these types of short-pulse laser systems the short pulse generation and the amplification is carried out at two different wavelengths. The seed pulse is generated in a dye laser system at 497 nm. After frequency doubling the short pulse is amplified in a KrF excimer amplifier in two passes. At the output laser pulses with high temporal and spatial quality with 500 fs pulse duration at 248 nm are available. The energy of the beam was concentrated on the silicon wafer by a quartz lens. The energy density on the surface was varied by z-scan. The femtosecond UV pulses were linearly polarized. The energy of the beam was 3 mJ with 10-20% amplified spontaneous emission (ASE) content. The pulse duration of the ASE was ~ 10 ns.

Two types of silicon wafers were irradiated by short UV laser pulses with the same parameters. The diameter of the wafers was 100 mm and the thickness was 0.5 mm. The resistivity of the n-type silicon with orientation (100) and of the p-type silicon with (111) orientation was $7.5 \Omega\text{cm}$, and $10 \Omega\text{cm}$, respectively. The wafers were mounted on a two axis translation stage and were irradiated in air environment. The samples were put out to an average energy density 1.44 Jcm^{-2} , 1.15 Jcm^{-2} , 0.83 Jcm^{-2} , 0.59 Jcm^{-2} , 0.28 Jcm^{-2} and 0.16 Jcm^{-2} with pulse numbers 1, 3, 5, 10, 15, 40, 70, 100, 170, 300 and 1000. The created surface structures were examined with a Hitachi S-4700 Scanning Electron Microscope (SEM).

EXPERIMENTAL RESULTS AND DISCUSSION

Figure 1 shows the SEM picture of the created surface on Si (100) at 1.44 Jcm^{-2} after 5 pulses. The diameter of the spot is $\sim 470 \mu\text{m}$. In the center of the irradiated spot a damaged area takes place with a diameter 20-100 μm . In the center of this damaged area a crater is present with $< 10 \mu\text{m}$ diameter. Outside of the damaged area a concentric periodic structure appeared at a certain distance from the crater. Between the crater and the first circle the structure is radial. The period of the concentric structure is increasing from $\sim 1.1 \mu\text{m}$ to $\sim 3.3 \mu\text{m}$ from the center to the edges. The width of the created grooves also increases along the radial direction.

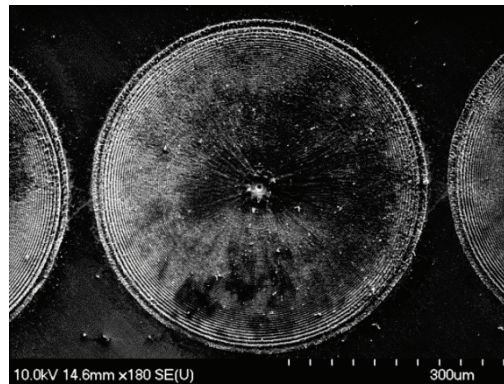


FIGURE 1. SEM image of the irradiated surface of Si (100) sample at 1.44 Jcm^{-2} after 5 pulses.

Figure 2(a)-(c) shows the formed periodic part of the surface structure after 5 pulses with fluences 1.44 Jcm^{-2} , 0.59 Jcm^{-2} and 0.16 Jcm^{-2} on Si (100). It can be observed that in case of lower energy densities the concentric periodic structure appears only in the edge of the irradiated surface.

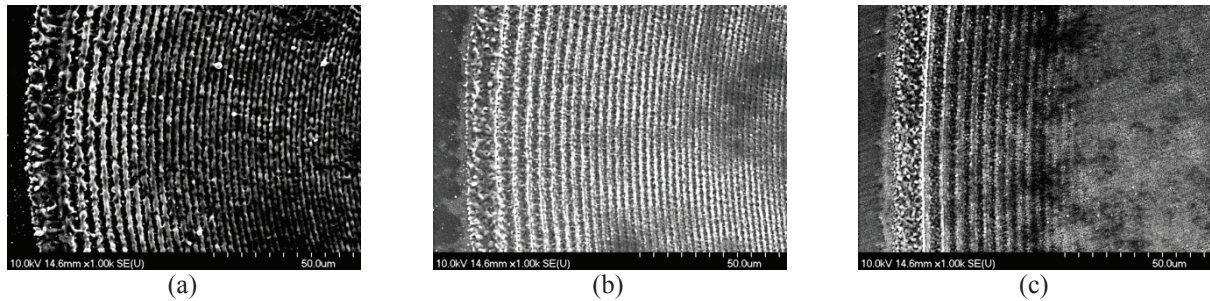


FIGURE 2. SEM image of irradiated surface of Si (100) after 5 pulses at 1.44 Jcm^{-2} (a), 0.59 Jcm^{-2} (b) and 0.16 Jcm^{-2} (c)

Figure 3(a)-(c) shows the evolution of the periodic part of the structure at 0.83 Jcm^{-2} fluence after pulses number of 5, 40 and 300. At higher pulse numbers the repeated machining of the irradiated surface leads to a granular structure. It is conspicuous that melting effects because of the ASE and oxidation because of the air environment take part in the cause of the granular structure, however oxidation effects are significant only for long pulses and lower energy densities. Figure 3d shows the global picture of irradiated Si (100) surface with 0.83 Jcm^{-2} fluence after 1000 pulses. The resulting surface can be divided into three major parts; an oxidation/amorphization area as an outer ring of the irradiated surface, a recrystallized rim area surrounding the irradiated spot and the irradiated spot with the periodic/granular structure. The source of the recrystallized rim could be the melted part of the sample from the edges of the beam spot. Surface acoustic waves can deliver the melted matter in radial direction.

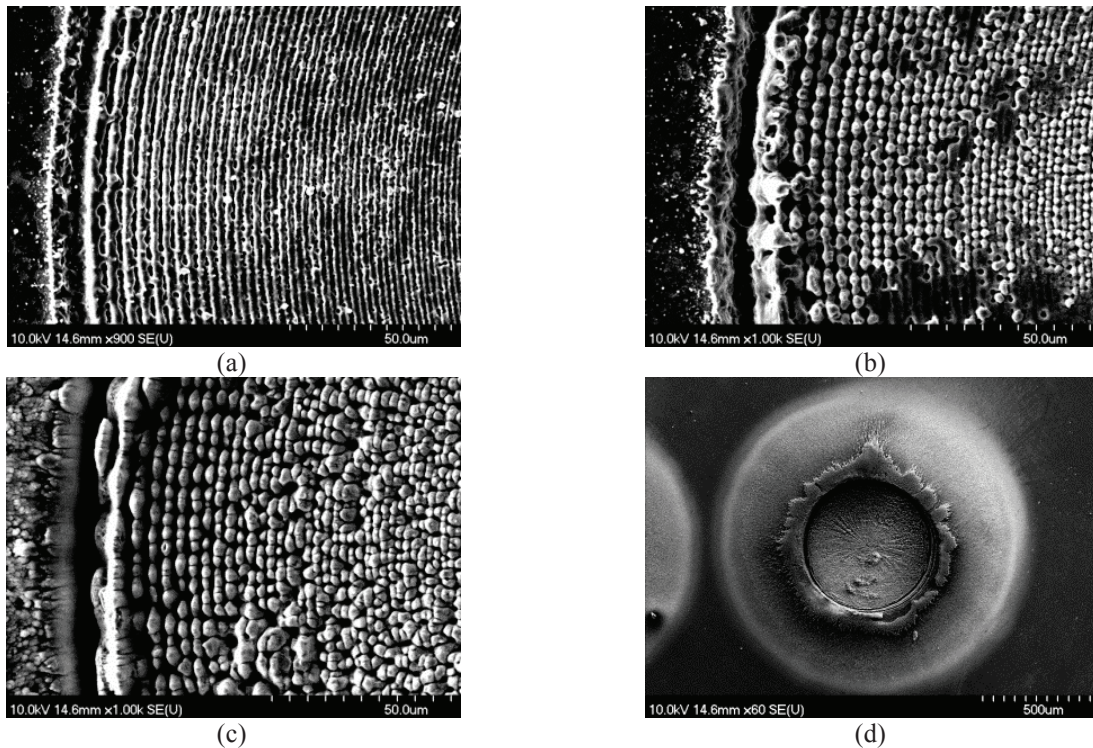


FIGURE 3. SEM images of the evolution of the periodic part of the structure on Si (100) at 0.83 Jcm^{-2} fluence after pulses number of 5 (a), 40 (b), 300 (c) and global picture of the processed surface after 1000 pulses (d)

Micromachining of Si (111) shows some difference compared to the Si (100) wafers. Figure 4 shows the formed surface structures of the Si (100) and Si (111) with 0.83 Jcm^{-2} after 40 pulses.

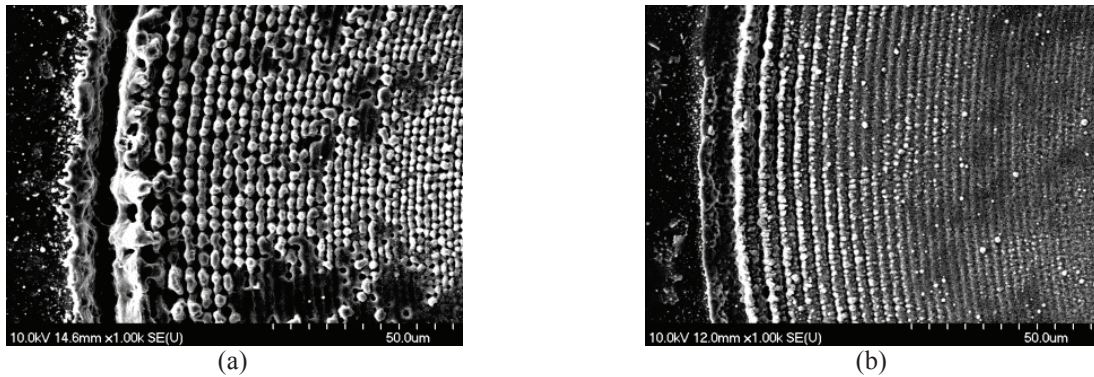


FIGURE 4. SEM image of Si (100) (a) and Si (111) surface after 40 pulses at 0.83 Jcm^{-2} .

The Si 100 sample already has a granular structure where in case of the Si 111 the periodic structure is still well defined. The cause of this effect could be the different material properties of the two types of silicon. However, we cannot disregard that the beam parameters were unfortunately slightly different in the two cases. The ASE content of the overall pulse energy was 20% for the Si (100) wafer and it was 10% for the Si (111) wafer. This circumstance can explain the difference of the state of the melting process.

The formation of the periodic microstructure seems different from the processes described in the literature of LIPPS (see introduction), since there is no relation between the direction of the periodic structure and the beam polarization vector. The silicon wafers were positioned close of the focal plane of the focusing lens. The direction of the created structure together with the variation of the periodicity in the radial direction and with varying spot size imply that the reason for the created structure is the Fresnel-diffraction of the beam near the focal plane. The idea of the periodic structure caused by Fresnel-diffraction is further supported by the fact that the applied laser beam was not Gaussian, the near-field beam profile was flat-topped where the edges of the beam was determined by an iris diaphragm. Unfortunately the spatial intensity distribution of the beam in the plane of the sample was not recorded during this experiment. However, in case of a similar experiment, the intensity distribution was recorded with a CCD camera and it is shown in Figure 5. It can be seen that the beam has a quite similar distribution to the formed surface structure in Fig. 1.

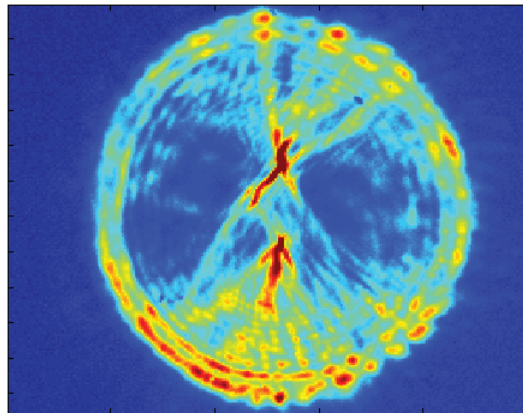


FIGURE 5. Typical intensity distribution of the UV femtosecond laser beam near the focal plane. with clearly visible Fresnel diffraction pattern.

CONCLUSION

Laser induced surface structures were formed on Si (100) and Si (111) wafers with 500 fs UV laser pulses. The created structure has a periodic part with a spatial period between $1.1 \mu\text{m}$ to $3.3 \mu\text{m}$ in a concentric organization. The origin of this type of structure is the inhomogeneity of the beam profile in the plane of the sample caused by Fresnel diffraction. UV femtosecond pulses seems to have a great advantage in this type of machining. The short wavelength makes possible to create μm sized structures and strong absorption (even in materials which are

transparent for wavelength of visible or near IR laser pulses). On the other hand the sub-ps pulse duration minimizes the heat affected zone. This type of machining has the advantage of high precision and reproducibility.

Further investigations of nano- and micromachining of silicon by femtosecond UV pulses is highly anticipated. The reduction of the spatial period by the set of proper diffraction image on the sample and determination the role of the ns ASE are the main perspectives of the future researches.

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REFERENCES

- [1] C. R. Phipps, *Laser Ablation and its Applications* (Springer, New York, 2007).
- [2] B. Rethfeld, K. Sokolowski-Tinten, D. VonDerLinde, S. I. Anisimov, *Appl. Phys. A* **79**, 767–769 (2009).
- [3] P. C. Becker, H. L. Fragnito, C. H. B. Cruz, R. L. Fork, J. E. Cunningham, J. E. Henry, C. V. Shank, *Phys. Rev. Lett.* **61**, 1647–1649 (1988).
- [4] W. H. Knox, C. Hirlimann, D. A. B. Miller, *Phys. Rev. Lett.*, **56**, 1191–1193 (1986).
- [5] J. Bonse, J. Krüger, S. Höhm, and A. Rosenfeld, *J. Laser Appl.* **24**(4), 042006 (2012).
- [6] T. H. Her, “Femtosecond-laser-induced periodic self-organized nanostructures” in *Comprehensive Nanoscience and Technology, Vol. 4*, edited by G. Scholes and G. Wiederrecht, (Elsevier, 2010) Chapter 4.10.
- [7] M. Huang, Y. Cheng, and Z. Xu, *Ann. Phys. (Berlin)* **525**(1–2), 74–86 (2013).
- [8] A. Vorobyev and C. Guo, *Laser Photon. Rev.* **7**(3), 385–407 (2013).
- [9] R. Buividas, M. Mikutis, and S. Juodkakis, *Prog. Quantum Electron.* **38**(3), 119–156 (2014).
- [10] J. Bonse, S. Baudach, J. Krüger, W. Kautek, and M. Lenzner, *Appl. Phys. A: Mater. Sci. Process.* **74**, 19-25 (2002).
- [11] T. J.-Y. Derrien, R. Koter, J. Krüger, S. Höhm, A. Rosenfeld and J. Bonse, *J. Appl. Phys.* **116**, 074902 (2014)
- [12] F. Costache, S. Kouteva-Arguirova, and J. Reif, *Appl. Phys. A: Mater. Sci. Process.* **79**, 1429 (2004).
- [13] V. S. Makin, Yu. I. Pestov, R. S. Makin, and A. Ya. Vorobyev, *J. Opt. Technol.* **76**, 555-559 (2009).
- [14] M. Huang, F. Zhao, Y. Cheng, N. Xu, and Z. Xu, *ACS Nano* **3**, 4062-4070 (2009).
- [15] J. Bonse, A. Rosenfeld, and J. Krüger, *J. Appl. Phys.* **106**, 104910 (2009).
- [16] E. G. Gamaly, *Appl. Phys. A* **101**, 205 (2010)
- [17] N. Medvedev, Z. Li, and B. Ziaja, *Phys. Rev. B* **91**, 054113 (2015)
- [18] J. Reif, F. Costache, M. Henyk, and S. V. Pandelov, *Appl. Surf. Sci.* **891**, 197–198, (2002).
- [19] D. Dufft, A. Rosenfeld, S. K. Das, R. Grunwald, and J. Bonse, *J. Appl. Phys.* **105**, 034908 (2009).
- [20] G. A. Martsinovskii, G. D. Shandybina, D. S. Smirnov, S. V. Zaboltnov, L.A. Golovan, V. Y. Timoshenko, and P. K. Kashkarov, *Opt. Spectrosc.* **105**, 67-72 (2008).
- [21] E.V. Golosov, A. A. Ionin, Y. R. Kolobov, S. I. Kudryashov, A. E. Ligachev, S. V. Makarov, Y. N. Novoselov, L. V. Seleznev, D. V. Sinitsyn, *Appl. Phys. A* **104**: 701-705 (2011).
- [22] A. A. Ionin, S.I. Kudryashov, S.V. Makarov, L.V. Seleznev, D.V. Sinitsyn, E.V. Golosov, O.A. Golosova, Yu.R. Kolobov, A.E. Ligachev, *Quantum Electronics* **41**, 9, 829-834 (2011).
- [23] J. Ihlemann, B. Wolff, and P. Simon, *Appl. Phys. A* **54**, 363-368 (1992)
- [24] S. Szatmári, F. P. Schäfer, *Opt. Commun.* **68**, 196-202 (1988)
- [25] Szatmári S, Marowsky G and Simon P *Femtosecond Excimer Lasers and their Applications*, Landolt Börnstein New Series VIII/1B edited by G Herziger et al (2007), pp 215-53.