Baku State University: Journal of Physics & Space Sciences, **2024**, v 1 (2), p. 63-69

*journal homepage: http://bsuj.bsu.edu.az/en*

# Laser ablation Si thin films

Vusal U. Mammadov*

*Baku State University, Baku, Azerbaijan*

Received 12-Apr-2024; Accepted 14-May-2024

---

**Abstract**

The processes of ablation of the surface of single-crystal silicon wafers and the properties of materials obtained because of silicon ablation under irradiation with a scanning beam with a wavelength of λ = 1062 nm were studied with varying laser radiation power and scanning modes. The range of beam scanning modes in which $SiO_2$ layer growth is observed has been established. Starting from a scanning speed of 2000 mm/s, the formation of silicon oxide stops, and because of the destruction of silicon, nanometer-sized silicon particles are formed. In this case, the destruction of silicon is accompanied by sounds of different frequencies, depending on the scanning speed.

*Keywords*: thin films; silicon; laser ablation; crystal, optical properties
*PACS*: 71.55.Gs; 78.20.-e; 79.20.Eb; 85.30.-z; 85.60.Dw

---

## 1. Introduction

The rapid development of lasers with ultrashort pulses opens up new possibilities for precision processing of materials, in particular silicon [1–3]. Due to its thermodynamic, physicochemical and semiconductor properties, silicon is widely used in micro- and nanoelectronics, sensor technology, biomedicine, etc. In this regard, at present, interest in the effect of laser radiation on silicon and the SiO2/Si system (a layer of silicon dioxide on the surface of a single-crystal silicon wafer) is continuously growing [4]. It is known that when laser pulses impact the surface of crystalline

**Corresponding author*. Tel.: +994-50-664-63-84
*E-mail address*: mammadovv@gmail.com; *ORCID ID*: 0009-0007-8084-3436.

solids and, in particular, a single crystal of silicon, the generation of structural defects in the surface layer is possible - vacancies and interstitial silicon atoms [5], the concentration of which during laser exposure can change compared to the original by several orders of magnitude and reach values of 1019−1021 $cm^{-3}$ [6]. The combination of extended defects into larger defects (in networks of dislocations or accumulation of pores) can initiate the formation of microcracks and destruction of the surface even before the start of melting. In [7], solid-phase destruction of the silicon surface under the influence of laser pulses in air and vacuum was studied. In another work by the same authors [8], it was found that when silicon surfaces are exposed to laser pulses with an intensity above the silicon melting threshold, particle emission occurs. The work on microstructuring the $SiO_2$/Si system shows the effect of laser irradiation of the system with a pulsed laser on the appearance of local plastic deformation of the silicon surface under silicon oxide. At the same time, upon transition to the scanning mode of a pulsed laser photon beam over the surface of the $SiO_2$/Si system, the mechanism of the appearance of local melting and its development with increasing irradiation power was demonstrated. This paper presents the results of a study of the processes of destruction of a single-crystalline silicon wafer as a result of irradiation by a laser operating in high-frequency scanning mode with a high irradiation energy density.

## 2. Materials and Methods

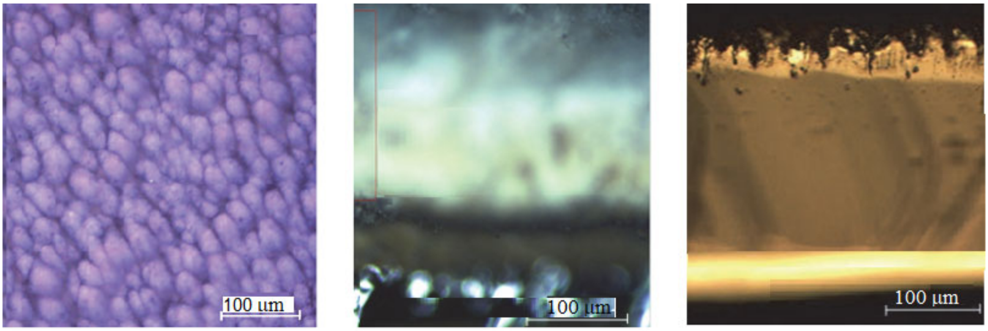
The studies were carried out on industrial silicon wafers KDB-10 (111), KEF-4.5 (100) with a layer of native silicon oxide several tens of nanometers thick and a $SiO_2$ layer thickness from 120 to 300 nm, grown by thermal oxidation. Irradiation was carried out using the MiniMarker 2 laser system. This system includes a pulsed laser with a wavelength λ = 1062 nm. Nominal laser output power 20 W, pulse duration 100 ns. The pulse repetition rate can be adjusted from 20 kHz to 100 kHz. The nominal energy per pulse at a maximum output power of 20 W is 1.0 mJ. Control of the scanning speed and the amount of overlap of scanning lines is possible in software mode. The scanning speed (v) varies from 10 mm/s to 4000 mm/s. The density of scanning line overlap (Ppr) varies from 100 lines/mm to 4000 lines/mm. Control of experimental samples was carried out using an optical microscope of the Axio Imager A1m type from Carl Zeiss, equipped with a high-resolution digital video camera. All experiments were carried out in the focused laser beam mode, when the diameter of the irradiation spot on the substrate was 50 µm. The substrate surface temperature and its area distribution were assessed in real time using a FLIR SC7000 series thermal imager.

## 3. Results and Discussions

The results of microstructuring of the $SiO_2$/Si system are influenced by factors such as radiation power, radiation energy density, pulse repetition frequency and duration, beam scanning speed, and the amount of overlap of the irradiation area by scanning lines. Based on this, at the initial stage, studies were carried out related to the choice of modes for scanning a laser spot on the irradiated area of the substrate (irradiation area 5 × 5 mm2, spot diameter 50 μm), since such data were absent in the literature. As a result of this stage of work, the laser beam scanning modes were selected: scanning speed – 100 mm/s, amount of scanning line overlap – 1000 lines/mm.

At the first stage of the work, the task was to study the influence of laser radiation power on the process of ablation of the surface of a silicon wafer under the scanning modes selected above. Taking into account the selected scanning and overlap speeds, the laser pulse duration was 100 ns at a repetition rate of 50 kHz. The laser radiation power varied from 2 to 20 W. Studies have shown that the minimum laser power at which silicon ablation begins is 4 W. At a power of 8 W or more, an increase in height and the appearance of a layer of white substance on the irradiated surface are observed. Etching such samples in a solution of hydrofluoric acid (HF) leads to rapid and complete etching of the resulting layers, which confirms the assumption of the oxidation of silicon particles in oxygen plasma and the formation of a $SiO_2$ layer on the surface of the irradiated region. A microphotograph of the surface of a section of a silicon wafer irradiated by a scanning beam of a fiber laser at a power of 10 W (Fig. 1, a).

As can be seen in the microphotograph of the chip (Fig. 1, b), the thickness of this layer can exceed 470 μm. The layer has a white color, a loose structure and is removed from the surface in the form of a fine powder with tweezers, filter paper or a cotton swab. Infrared (IR) transmission spectra of the substance formed during silicon ablation are shown in Fig. 2. Intense transmission bands were found in these spectra, located in the region of wave numbers 527, 612, 980 and 1130 $cm^{-1}$. The appearance of these bands indicates the formation of nanoparticles of the SiO2 or SiOx phase (x = 1.5–2) on the surface of the irradiated area of the substrate. In the spectra of the samples, in addition to the above vibration modes belonging to the oxide phase, transmission bands appeared in the regions of 667 and 1105 $cm^{-1}$, which can be attributed to the SixOyNz oxynitride phase or, most likely, to a trace of adsorbed water. Thus, the IR transmission spectra confirm the chemical composition of the condensate and its origin, associated with the oxidation of ejected silicon particles in the plasma plume and their subsequent condensation on the surface with the formation of a $SiO_2$ layer.




a. в. c.

**Fig. 1.** Photo of experimental samples: the surface of the irradiated area of the silicon wafer after scanning the laser beam (a); fragment of a chip of the irradiated area (b); fragment of a chip after etching a SiO2 layer in a HF solution and washing the substrate in a stream of water (c)

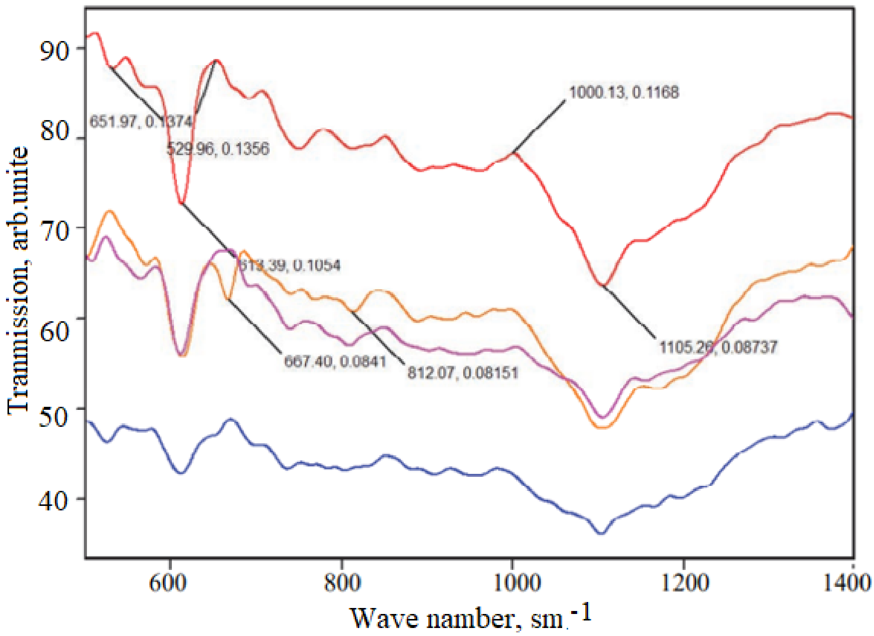



**Fig. 2.** IR transmission spectra of the substance formed during silicon ablation. The curves correspond to the scanning speed: yellow – 8750 mm/s; red – 3000 mm/s; pink –1000 mm/s; blue – 100 mm/s.

In Fig. 1b, a lower layer with reflections from the surface of individual crystal protrusions is also observed, which is a damaged layer of the single-crystal substrate. The middle (dark gray) layer consists of small particles of silicon formed as a result of layer-by-layer crushing (cracking) of the single-crystal substrate. At the same time, as follows from Fig. 3, the temperature of the irradiation area of the silicon wafer is about 1000 ºC, i.e. significantly lower than the melting point of silicon (1423 ºC). This is also evidenced by the fact that after etching experimental samples in a solution of hydrofluoric acid (HF) (Fig. 1, c), the $SiO_2$ layer is completely removed, and small silicon particles, after washing in a stream of water, settle in the solution. On a chipped substrate after etching, the surface of the damaged layer has a cracked structure without any traces of silicon melting. However, the results of the following experiment indicate that in addition to the amount of energy received by the material as a result of exposure to the laser pulse and the distribution of this energy in the irradiated sample, the method of energy transfer to the crystal is also an important factor affecting the ablation mechanisms and the parameters of the ejected material.

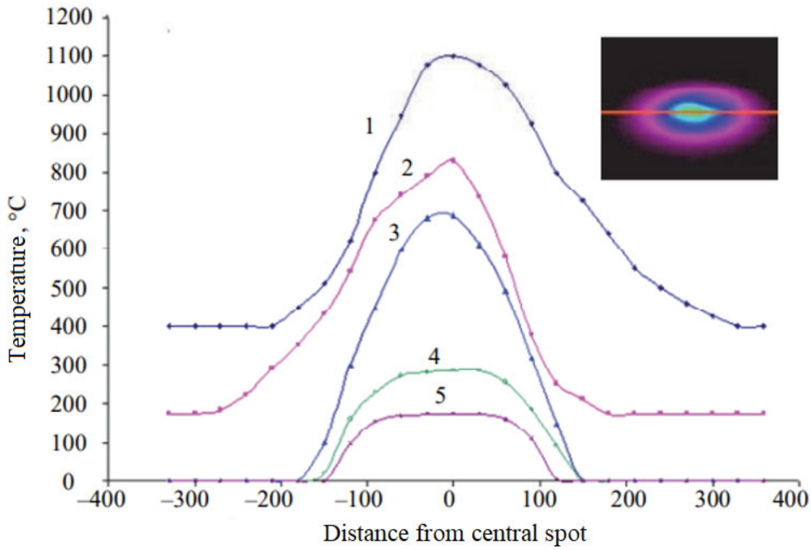



**Fig. 3.** Temperature profiles of the spot along the scanning direction at different powers (1 – 20 W; 2 – 16 W; 3 – 12 W; 4 – 8 W; 5 – 4 W). The inset shows a thermogram of the irradiation zone.

To establish a more accurate range of beam scanning modes in which the growth of the $SiO_2$ layer is observed, the beam scanning speed was chosen as a variable parameter that ensures a smoother change in the total number of pulses incident on the irradiated area of the substrate. The microstructuring process was carried out under the same laser operating conditions as in the previously described experiment. Only the pulse repetition rate was changed (99 kHz instead of 50 kHz). As before, the area of the irradiation areas was 5×5 $mm^2$, the diameter of the laser spot was 50 µm. When irradiating the surface of a single crystal of silicon with a laser, only the scanning speed of the laser beam changed in the speed range from 10 mm/s (when the first silicon oxide clusters appear on the irradiated area) to 4000 mm/s. In Fig. 4 shows microphotographs of the surface of experimental samples after laser microstructuring of both the $SiO_2$/Si system and silicon wafers at different scanning speeds.

It was found that the maximum amount of $SiO_2$ is formed in the irradiated area at scanning speeds from 100 to 300 mm/s. Starting from a scanning speed of 2000 mm/s, a layer of silicon nanoparticles is formed on the surface. The thickness of this layer increases with increasing irradiation time. The course of the ablation process is not affected by either the electrical properties and crystallographic orientation of the single crystal, or the presence or absence of a $SiO_2$ film on the surface.

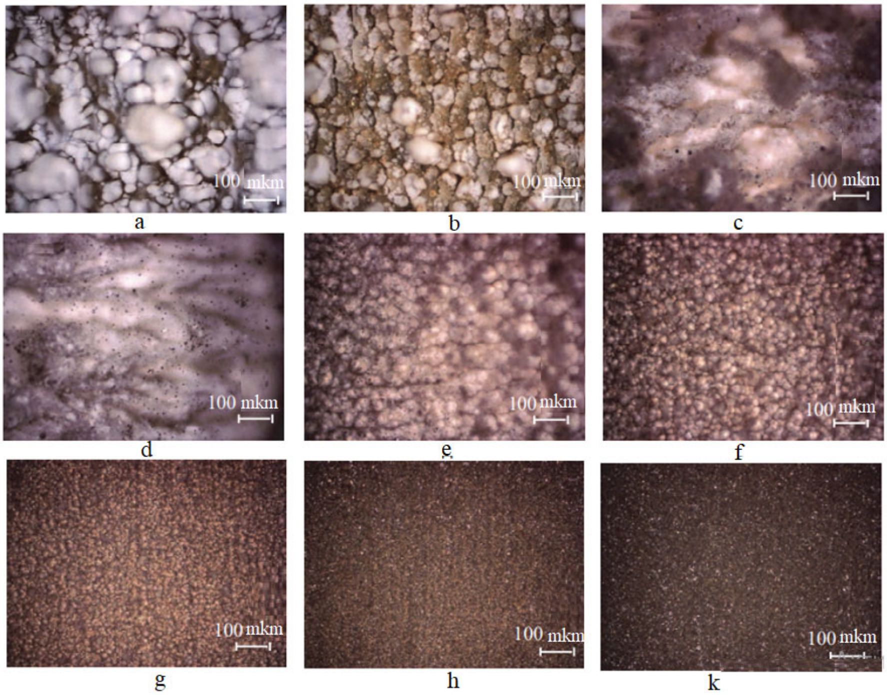



**Fig. 4.** Microphotographs of sections of the scanning area obtained at a power density of $1.02 \cdot 10^6$ W/cm$^2$ with different scanning speeds: 10 mm/s (a); 50 mm/s (b); 100 mm/s (c); 300 mm/s g); 600 mm/s (d); 1000 mm/s (e); 2000 mm/s (w); 3000 mm/s (w); 4000 mm/s (i). In all cases, the overlap density Ppr = 1000 lines/mm, f = 99 kHz

## 4. Conclusions

The paper presents the results of a study of the process of laser ablation of silicon when irradiated by a scanning laser beam with a wavelength λ = 1062 nm. It has been shown that the process of silicon ablation under these conditions occurs without silicon melting. The determining factor in this process is the total number of laser pulses incident on the irradiation area. Irradiation modes that affect the composition of the resulting ablation products have been determined. The process of silicon ablation with the subsequent formation of silicon dioxide in the above modes is not affected by either the silicon parameters (conductivity type, orientation and resistivity) or the presence or absence of a silicon dioxide layer on the surface of the silicon substrate.

## References

[1] Sanchez, C.; Julian, B.; Belleville, P.; Popall, M., Application of Hybrid OrganicInorganic Nanocomposites. Journal of Materials Chemistry 2005, 15, 3559-3592.

[2] Lukowiak, A.; Strek, W., Sensing Abilities of Materials Prepared by Sol-Gel Technologies. Journal of Sol-Gel Science and Technology 2009, 50, 201-215.

[3] Arkles, B., Commercial Applications of Sol-Gel Derived Hybrid Materials. Materials Research Society Bulletin 2001, 26, (5), 402-408.

[4] Galarneau, A.; Iapichella, J.; Bonhomme, K.; Di Renzo, F.; Kooyman, P., Controlling the Morphology of Mesostructured Silica by Psuedomorphic Transformation: A Route to Towards Applications. Advanced Functional Materials 2006, 16, (1657-1667).

[5] Nakanishi, K.; Nunez, O.; Tanaka, N., Preparation of Monolithic Silica Columns for High Performance Liquid Chromatography. Journal of Chromatography A 2008, 1191, 231-252.

[6] Kato, M.; Sakai-Kato, K.; Toyo'oka, T., Silica Sol-Gel Monolithic Materials and Their Use in a Variety of Applications. Journal of Separation Sciences 2005, 28, 1893- 1908.

[7] Nakanishi, K.; Tanaka, N., Sol-Gel with Phase Separation: Hierarchically Porous Materials Optimized for High Performance Liquid Chromatography Separations. Accounts of Chemical Research 2007, 40, 863-873.

[8] Yuan, Z. Y.; Su, B. L., Insights into Hierarchically Meso-Macroporous Structure Materials.. Journal of Materials Chemistry 2006, 16, 663-667.