Laser-micromachining silicon three-dimensional structures, tunnels, and cavities

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Abstract: A laser-micromachining technique has been developed to make tunnels and cavities in Si under SiO₂ or Si₃N₄ films. Based on the laser-induced Cl₂ etching of Si and high chlorine Si:SiO₂ and Si:Si₃N₄ etch-rate ratios, tunnels with length of up to 3 mm and cavities of 100×100 μm² were successfully fabricated in SiO₂/Si bilayered samples. Similarly, cavities of 50×50 μm² were also fabricated in Si₃N₄/Si samples.

Résumé: Nous avons développé une méthode de micro-usinage au laser permettant la réalisation de tunnels et de cavités dans un substrat de Si, sous des couches de SiO₂ ou de Si₃N₄. Cette technique est fondée sur la gravure du Si par Cl₂ et exploite les valeurs élevées des rapports de sélectivité Si:SiO₂ et Si:Si₃N₄. Des tunnels de longueur allant jusqu'à 3 mm et des cavités de 100×100 μm² ont été usinés dans des échantillons bicouches SiO₂/Si. Des cavités de 50×50 μm² ont également été réalisées avec succès dans des substrats Si₃N₄/Si.

1. Introduction

Conventionally, silicon micromachining is achieved using the photolithographic process, followed by wet anisotropic etching or reactive ion etching (RIE) [1, 2]. While successfully evolving from microelectronic fabrication technologies, these conventional techniques have some inherent limitations, such as 2D patterning and, in the case of wet etching, a dependence on the crystallographic orientation of the substrates [1, 2]. When the sacrificial layer technique is used, complex multi-patterning, multi-layering processes and lift-off etching are involved [1, 2]. Laser micromachining, which has provoked great interest over the last decade [3], has none of these shortcomings but rather several benefits that make it an attractive alternative technique [4–6]. Here the laser micromachining of silicon refers to the laser-induced chlorine etching of silicon. Since it is a computer-controlled, truly three-dimensional (3D) process permitting maskless patterning, applications in the emerging field of microelectromechanical systems (MEMS) have been explored [7–10]. Two important aspects of the laser micromachining of silicon are the transparency of silicon dioxide (SiO₂) and silicon nitride (Si₃N₄) to the Ar⁺ laser and the high chlorine Si:SiO₂ and Si:Si₃N₄ etch-rate ratios [11]. These properties permit silicon to be patterned under SiO₂ or Si₃N₄ layers, yielding buried structures. In this way, tunnels and cavities may be fabricated [11–13]. Such microstructures may be applicable to microfluidic devices and freestanding parts of MEMS. In this paper, after presenting our laser-micromachining system, we report on the laser-induced Cl₂ etching of bare Si and on the fabrication of tunnels and cavities on SiO₂/Si and Si₃N₄/Si bilayered samples.

2. Laser-micromachining system and experimental techniques

A schematic of the laser-micromachining system is shown in Fig. 1. It consists of a cw Ar⁺ laser, an optical system that is used to project the laser beam into a stainless steel reaction cell, a gas distribution system, and computer-controlled x-y translation stages. The Ar⁺ laser may be set to three operation configurations in terms of wavelength and laser output power; when working at 488 nm, 514 nm, and multimode modes, the laser provides maximum powers of 1.5, 2, and 5 W, respectively. The optical system comprises mirrors, a dual wavelength quarter-wave plate (for 488 and 514 nm), and a microscope with a long-working-distance objective (25X, 0.31 NA). The quarter-wave plate is used to eliminate the polarization effect on etched structures [14], although the improvement is limited for the multilayer operation. A beam splitter is inserted in the beam path to permit an operator to monitor the etching process on a video screen.

The laser TEM₀₀ beam, which was projected and focused by the optical system, entered the reaction cell through a quartz window to irradiate the substrate surface. The laser beam spot diameter at the focal plane of the microscope objective was measured using the knife-edge technique [15] and found to be 7 μm. The focus position could be adjusted by moving the z-direction translation stage so that the energy density at the surface could be modulated and controlled. The laser direct-writing is controlled by the x-y translation stages, with a maximum speed of 16 mm s⁻¹ and a spatial resolution of 0.1 μm.

After pumping down to 10⁻³ Torr (1 Torr = 133.3 Pa), the reaction cell and the gas distribution tubes were purged using N₂ and Ar gases. The reaction cell could be heated to accelerate the outgassing. The cell was then filled with high purity (99.99%) Cl₂; at pressures ranging from 100 to 500 Torr. The silicon surface to be patterned was moved vertically (z-direction) to be set in the focal plane of the objective and the laser was...
turned on at a sufficiently high power to heat the local surface to its melting temperature (1410°C). This melting threshold must be reached in the micromachining process to obtain the highest etching rates [6]. The localized melted zone defines a reaction area where the dissociation of the reactive chlorine molecules occurs. It must be emphasized that the dissociation of the chlorine molecules is pyrolytically controlled at the heated surface. No photolytic effect takes place in the gas phase where Cl₂ is almost totally transparent to the Ar⁺ laser wavelengths [3]. The dissociation of the chlorine molecules results in the generation of free chlorine atoms (radicals) that react with the silicon to form volatile SiCl₂ (x ≥ 2) compounds [6]. By moving the surface in the focal plane horizontally, the melted zone and, therefore, the etching can be displaced so that direct-writing patterns can be generated. The spatial resolution of the process almost corresponds to the dimension of the melted zone, which may be smaller than the beam spot size due to the Gaussian shape of the TEM₀₀₀ beam [16–18].

Two sets of substrates were used. The first consisted of <100> Si wafers. These samples are first cleaned by successive immersing in hot trichloroethane (TCE), acetone, and isopropyl alcohol, then rinsed in deionized (DI) water. For the substrates on which the etching was bare silicon, the native oxide was removed using a 1:10 buffered HF solution for 30 s. After removal of the native oxide for the substrates on which the buried structures were machined, dry oxidation (O₂ + N₂ atmosphere, 1200°C) of silicon was carried out. SiO₂ films with thicknesses of 0.2 and 0.3 μm were grown. The second set consisted of silicon wafers covered with 0.7 μm thick PE-CVD Si₃N₄ films whose compositions have been verified to be stoichiometric (3:4), as measured by X-ray photoelectron spectroscopy (XPS). Note that silicon nitride films with 3:4 stoichiometry are transparent to Ar⁺ laser [19]. The samples were cleaned using procedures similar to those mentioned above, except for the buffered HF-stripping step.

For etching beneath the SiO₂ or Si₃N₄ films, arrival and renewal of the Cl₂ gas at the reaction zone must be assured. Hence, prior to the introduction of the Cl₂ gas into the cell under vacuum, removal of the surface overlayer was performed using the Ar⁺ laser beam to form an opening of 200 × 200 μm². Since the SiO₂ or Si₃N₄ films are transparent to the Ar⁺ laser, the high-power beam was absorbed by the silicon substrate to form a molten layer that vaporized. The vaporization process may create a recoil pressure on the liquid layer and expel the molten silicon, which can break the overlayer leading to the removal of the SiO₂ or Si₃N₄ films. Another process that may play a role in this removal is the formation of volatile species, such as SiO₂, at high temperature. The direct-writing of the tunnels or cavities under the SiO₂ or Si₃N₄ was then carried out by moving the beam, which was initially focused on the bare silicon, from the exposed silicon surface toward the covered area.

A scanning electron microscope (SEM) was used to examine the etched structures, as well as to measure the etch depths.

3. Results and discussion

The preliminary experiments were performed on bare silicon substrates. The etch rate depends on the laser power, Cl₂ pressure, and scanning speed in a complex way. Figure 2a shows a SEM microphotograph of some grooves etched at a Cl₂ pressure of 500 Torr and a scanning speed of 100 μm s⁻¹. As the laser power of the 514 nm radiation was increased from 0.8 to 1.6 W, the grooves became deeper, varying from 0.4 to 8.1 μm. The width of these grooves correspondingly varied from 1.5 to 4.4 μm. Switching to the multiline operation mode, the same behavior was observed but, for powers beyond 3.2 W, did not result in greater depths. This may be seen in Fig. 2b where the dependence of the etch depth on the laser power is given for both operations. The increase in the etched depth with power, followed by saturation, may be related to the increase in the melted proportion of the irradiated silicon [20]. When a continuous, sufficiently thick, melted layer is obtained, no further increase in the depth is observed since any further increase of the beam energy will primarily contribute to latent heat [3, 21]. In the multiline operation mode, etch rates as high as 4 × 10⁻² μm² s⁻¹ were obtained. This value is comparable to that obtained in other studies [6, 7, 11].

The laser machining of buried structures was performed using 514 nm radiation. Laser powers ranging from 0.9 to 1.4 W, Cl₂ pressures of 100–500 Torr, and scanning speeds of 10–100 μm s⁻¹ were used. Figure 3a shows a top view of tunnels etched under a 0.2 μm thick SiO₂ film at 500 Torr of Cl₂ and different scanning speeds and laser powers. These tunnels are 450 μm long and 3 μm wide. Tunnels up to 3.5 mm long were also successfully obtained. Figure 3b shows an enlargement of the etching starting points in the initially ablated area. Starting at the exposed silicon surface, the etching is then displaced toward the SiO₂/Si area (to the left, in Fig. 3b). This adopted architecture permits the continued supply of Cl₂.
gas to the reaction zone. It must be emphasized that, even at a pressure of 100 Torr and a scanning speed of 100 μm s⁻¹, no evidence of gas transport limitation into the tunnels was observed. There is enough Cl₂ to sustain the reaction in tunnels as long as 450 μm. Indeed, by simply performing an additional etch starting at one of the previously machined tunnels, an additional interlinking of tunnels can be made, Fig. 3b. The SiO₂ film covering the tunnels was then carefully examined using high-resolution SEM, no physical defects were found in the overlayer, though the optical properties of the film need further characterization.

Figure 4 shows a cleaved sample in which two tunnels were etched under 0.2 μm thick SiO₂ at a laser power of 1.4 W, a Cl₂ pressure of 500 Torr, and scanning speeds of 10 μm s⁻¹ (for the left tunnel) and 30 μm s⁻¹ (for the right tunnel). The tunnels etched at 30 and 10 μm s⁻¹ are 1.5 and 3.3 μm deep, and 2.6 and 2.8 μm wide, respectively.

By etching tunnels with several side by side paths (raster scan), with an overlap, it is possible to machine cavities. Figure 5 shows such a cavity of 190 × 100 μm² etched under a 0.2 μm thick SiO₂ layer at a Cl₂ pressure of 500 Torr, a laser power of 1.2 W, and a scanning speed of 30 μm s⁻¹. The spacing between each tunnel is 2 μm. It took about 3 min to complete this fabrication. If higher scanning speed is used, the fabrication time could be shorter. The supply of the reactant gas to the machined area was provided through the tunnel connected at the top right corner of the cavity where the raster scanning started. This tunnel began at an ablated square area as that shown previously (Fig. 3), and was 1.6 mm long. Note that the gas supply to the etching area was not restricted by the turning point seen at the upper right corner of the cavity.

Since Si₃N₄ is a material whose mechanical properties are also of interest for MEMS [1], it is of interest for machine-buried structures under Si₃N₄ films. Figure 6 shows a cavity of 50 × 50 μm² etched in silicon under a 0.7 μm thick Si₃N₄ film. The scan started at the upper left corner, where a hole
was first etched to supply the Cl\textsubscript{2} gas. The process was basically successful except that there is a small opening on one side of the square cavity. This may be due to the stress release of the Si\textsubscript{3}N\textsubscript{4} film. Further investigations are in progress to avoid such imperfections.

4. Conclusions

Tunnels and cavities were successfully machined in silicon under SiO\textsubscript{2} films. No defects were observed in the SiO\textsubscript{2} overlayers. The use of Si\textsubscript{3}N\textsubscript{4} instead of SiO\textsubscript{2} gave encouraging results.

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