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Laser-Enhanced Chemical Etching of Solid Surfaces

The laser-enhanced chemical etching of Si, SiO_2, Ta, and Te films with halogen-containing gases excited by a pulsed CO_2 laser and a continuous-wave (cw) Ar^+ laser has been studied. Detailed measurements of the etch rates as functions of the laser frequency, the laser intensity, and the gas pressure have been performed for some of the gas–solid systems. The enhanced surface reactions have been classified into three categories: those activated by the vibrational excitation of the etchant molecules, those with radicals generated by photodissociation, and those induced by laser excitation of solid substrates. Examples which illustrate the effects of laser radiation on these surface photochemical processes are given. Achievable etch rates and spatial resolutions for the various reaction mechanisms are also examined.

Introduction

In recent years, there has been increasing interest in developing new techniques for processing electronic materials in response to problems involved in the fabrication of microelectronic circuits and micromechanical devices whose configurations are rapidly increasing in complexity. The new techniques are based primarily on radiation-enhanced chemical interactions at gas–solid interfaces by ions, electrons or photons [1–8]. The energetic particles are capable of exciting gas-phase molecules, chemical species adsorbed on surfaces, and/or surface atoms, and can thereby influence surface chemical interactions. One class of such radiation-enhanced chemical techniques involves the utilization of laser photons with wavelengths ranging from the ultraviolet and visible to the infrared. Laser radiation is readily focused onto solid surfaces and therefore is well-suited to promoting surface reactions with high spatial resolution. In addition, the monochromaticity, coherence, and high photon flux of the laser light are highly advantageous. Indeed, dry chemical etching [5–11], doping of semiconductors [8], and chemical vapor depositions [8, 12–14] have been demonstrated using lasers. Laser-enhanced electrochemical plating and etching for the liquid–solid systems have also been reported [15].

In order to better understand the various reaction mechanisms involved in laser-enhanced chemical etching, we have studied a variety of gas–solid systems, including Si, SiO_2, Ta, and Te surfaces using various halogen-containing gases. A high-power pulsed CO_2 laser and a continuous-wave (cw) argon ion laser have been used. We have identified several important reaction mechanisms by means of systematic studies of the etch rates and surface reaction yields as functions of the laser wavelength, laser intensity, and gas pressure. In general, etching of a solid material by exposure to gas-phase molecules includes processes of dissociative chemisorption, reaction between adsorbed species and solid atoms, and desorption of product molecules. It is thus possible to use an ir laser to excite molecules into highly vibrationally excited states, thereby enhancing the process of dissociative chemisorption and subsequent surface reactions to form volatile products [7, 16]. The ir laser can also be utilized to promote multiple-photon-induced dissociation of gaseous molecules and thereby create reactive radicals for surface reactions [6, 16]. In certain instances, reactive fragments can be produced effectively by single-photon photolysis in the uv and visible spectral regions [9, 11]. In still another class of experiments, laser beams can be used directly to excite solid atoms for enhancement of surface chemistry. In such cases, surface etching occurs without excitation of the gaseous species [10, 17]. These various reaction mechanisms are illustrated in this paper and the achievable etch rates and spatial resolutions, when applicable, are discussed.

Experimental conditions

Two experimental apparatuses were used for our studies of the laser-enhanced chemical etching. Apparatus A consisted of a pulsed CO_2 laser and a stainless steel vacuum chamber

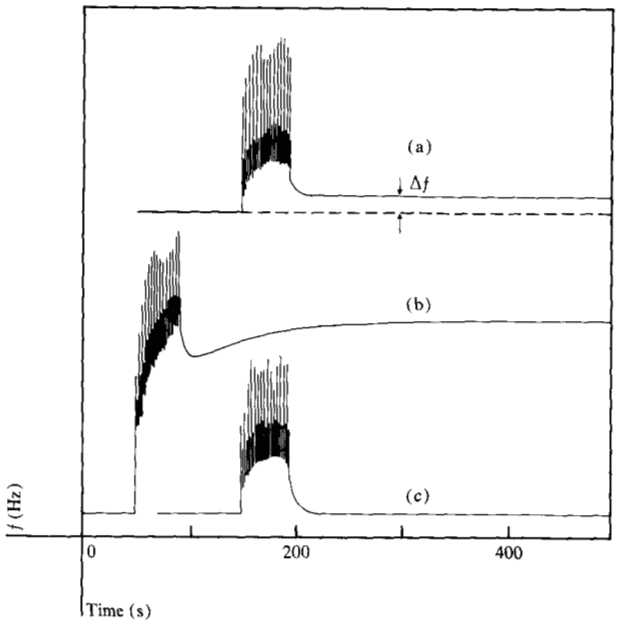

Figure 1 Frequency responses of the (a) Si, (b) Ta, and (c) SiO_2 microbalances in SF_6 excited by CO_2 laser pulses at 942.4 cm^{-1} with $I = 1$ J/cm^2, normal incidence, and $P = 133$ Pa. The net increases in frequency (Δf) due to 15 laser pulses were 30, 370, and 0 Hz, respectively.

equipped with a quartz-crystal microbalance (QCM). This apparatus was used for most of the results reported here and its detailed description has been published previously [16]. Briefly, the CO_2 laser-enhanced etching yields of the Si, SiO_2, Ta, and Te films (2 μm thick) on separate QCMs are measured from frequency changes of the microbalances, which have a sensitivity of $\approx 1.23 \times 10^{-8}$ g/Hz-cm^2. The QCM is maintained at 20°C by water cooling and can be positioned either parallel or perpendicular to the laser beam. For excitation, a Tachisto grating tuned TEA CO_2 laser is used. The laser, which is line-tunable in 9–11 μm, provides a pulse energy of 1.4 J with a pulse duration of $\approx$100 ns. The experimental procedure involves cleaning of solid surfaces by Ar ion bombardment, exposure of these surfaces to SF_6 gas at a given pressure or to XeF_2 gas under a steady-state-flow condition, and firing of laser pulses at 4-s intervals for a desired number of pulses. The net increase in the frequency of the QCM which corresponds to the net loss of solid atoms from the surfaces is recorded for analysis. All high-purity gases for the experiments were obtained from Matheson and PCR.

Apparatus B consisted of a cw Ar^+ laser (Spectra Physics) and a small stainless steel chamber with a sample holder. The laser, which is capable of providing 8.0 W of laser power at 514.5 nm and 1.9 W at 457.9 nm, is focused with a 12-mm focal length (f.l.) lens onto the solid substrate. This apparatus is used for the SiO_2–Cl_2 experiments. An Alpha-Step Profiler with a diamond stylus (Tencor Instruments) is utilized to obtain profiles of the etched features.

Results and discussion

• *Chemical etching activated by molecular vibrational excitation*

The typical frequency responses f of the Si, SiO_2, and Ta microbalances positioned perpendicular to the CO_2 laser beam using Apparatus A are shown in Fig. 1. The momentary increase in the frequency of the QCM during each laser pulse is due to a temporary temperature rise in the substrate caused by the laser pulse since the oscillation frequency of the quartz crystal is sensitive to temperature. In the absence of any reactive gas, the QCM returns to its original frequency $\approx$15 s after the laser pulses are fired. Thus, under the experimental conditions, the CO_2 laser radiation alone does not cause any significant removal of Si, SiO_2, or Ta atoms from the surfaces.

For the Si–SF_6 system, at a gas pressure $P = 266$ Pa excited with 1.0-J/cm^2 laser pulses at 942.4 cm^{-1}, we observe an increase in the frequency of the Si microbalance by $\Delta f = 2.3$ Hz/pulse, corresponding to an etching yield EY of 3.03×10^{14} Si atoms chemically removed per laser pulse. As shown previously [16], the total reaction yield is linearly proportional to the number of laser pulses and is strongly dependent on the laser wavelength. Namely, the Si–SF_6 reaction occurs only when SF_6 molecules are vibrationally excited; at laser intensity $I \leq 1$ J/cm^2, Si etching does not occur by pure laser excitation of the Si substrate in the absence of SF_6 molecular excitation. For $I = 0.1$–1 J/cm^2, $EY \propto I^{3.5}$, indicating that three or more photons are likely to be involved in promoting SF_6 molecules into high vibrational levels to overcome the activation barrier for reaction. The pressure dependence of the Si–SF_6 reaction yield at normal laser incidence further shows that in the low-pressure region EY increases with P; for $P \geq 266$ Pa, EY is essentially constant as long as the laser beam is not substantially attenuated by the gas along the optical path to the Si surface. This pressure dependence is due mainly to the gas-phase collision effect. Collisional deactivation can confine the vibrationally excited SF_6 molecules that are available to react with Si in a small region just above the Si surface.

The effect of gas-phase collisions on the lifetime of the chemically active states can have an important consequence on the spatial resolution of etched features. At $P = 266$ Pa, the mean free path λ_c for molecular collision is $\lesssim$0.1 mm and the vibrational relaxation time for SF_6 molecules via near-resonant vibrational energy exchange is $<$0.25 μs [18]. At this rate, the CO_2 laser-excited SF_6 molecules can be deacti-

vated within a radius of about 0.1 mm or less. As the gas pressure increases, λ_c decreases, *e.g.*, at 2.7×10^3 Pa, $\lambda_c \leq 10$ μm and at 1.3×10^4 Pa, $\lambda_c \leq 2$ μm. Consequently, with a CO_2 laser beam diameter d, the diameter of the etched area can be $\approx(d + 2\lambda_c)$, as illustrated in Fig. 2. The minimum diameter of the ir laser beam is limited by diffraction to about 10 μm. However, because of the collisional deactivation effect, directional etching of features only slightly larger than 10 μm can be obtained; see Fig. 2(b). Our measured Si etching yield is about 0.12 nm/pulse. With a commercially available CO_2 laser at a pulse rate of 100 pulses/s, an etch rate >0.7 μm/min is obtainable. Furthermore, as shown in Fig. 1(c), similar experiments for SiO_2 films do not yield significant etching of the solid. Thus, selective (differential) etching of Si *vs.* SiO_2 is possible using this method.

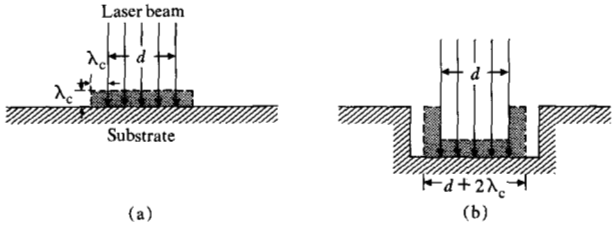

Figure 2 Expected directional etching of a substrate by vibrationally excited molecules promoted by an ir laser with beam diameter d (a) before and (b) after etching to some depth. Collisional deactivation can confine the chemically active molecules that are available to react with the substrate in a small region above the surface as indicated in the shaded area. λ_c is the mean free path for collisional deactivation determined by the gas pressure (see text).

The CO_2 laser-induced Ta–SF_6 reaction [Fig. 1(b)] is quite similar to that for Si–SF_6 except that more complicated reaction sequences are involved, including possible secondary surface reactions with SF_4, which is produced from the initial laser-induced SF_6 reaction with the Ta metal film [10]. The total Ta–SF_6 reaction yield at a given SF_6 pressure is higher than for the Si–SF_6 system. For example, with the CO_2 laser operating at 942.4 cm^{-1} and $P = 133$ Pa ($I = 1.0$ J/cm^2), $EY = 2.6 \times 10^{14}$ Si atoms/pulse for the Si–SF_6 system. Under the same conditions for the Ta–SF_6 system, $EY = 5.0 \times 10^{14}$ Ta atoms/pulse.

• *Chemical etching by photon-generated radicals*

Photodissociation of molecules to produce reactive radicals can be accomplished by multiple-photon excitation in the ground electronic state or by single-photon photolysis involving excited electronic states. The phenomenon of multiple-photon dissociation, in particular the dissociation of SF_6 molecules by intense CO_2 laser pulses, has been extensively studied in recent years [19]. For our SF_6 multiple-photon dissociation experiments, we used a focused CO_2 laser with an intensity $I \gtrsim 10$ J/cm^2 to produce SF_4 and F atoms. The SF_4 molecules are relatively inert to Si, but F atoms can react with the solid at room temperature. We have measured $\Delta f = 6$ Hz using 60 laser pulses at 942.4 cm^{-1} and 0.5 J energy focused about 1 mm above the Si surfaces in 200 Pa of SF_6. The focused laser beam is parallel to the surface and the Si substrate is not subjected to the laser irradiation. Under this condition, $EY \approx 1.3 \times 10^{13}$ Si atoms/pulse (*i.e.*, $\approx$0.005 nm/pulse) and it decreases as the distance between the focused laser beam and the surface increases. Even fluorine atoms generated several millimeters away from the surface can survive collisions with other gas-phase molecules and diffuse to the Si surface for reaction. This is in great contrast to the reaction of Si with vibrationally excited SF_6 molecules that can be chemically deactivated within a radius of <0.13 mm at 200 Pa. Similar experiments with the SiO_2–CF_3Br system have been reported by Steinfeld *et al.* [6]. It was shown that CF_3Br molecules could be dissociated by multiple CO_2 laser photons to generate CF_3 radicals, which reacted with SiO_2 to give $EY = 0.03$ nm/pulse.

For the laser chemical etching of SiO_2, we have chosen the method of single-photon photolysis of Cl_2 gas. It is known that chlorine molecules can be excited into repulsive electronic states which cause Cl_2 to decompose into Cl atoms in the 250–500-nm region [20]. By itself, Cl_2 is not reactive to SiO_2, and the quartz substrate is optically transparent in this spectral region. Using Apparatus B and the Ar^+ laser at 457.9 nm, with the 1.85-W laser power focused with a 12-mm f.l. lens, we are able to etch a quartz flat in 1.33×10^4 Pa of Cl_2 gas at the rate of $\approx$20 nm/min. The results are shown in Fig. 3. Also shown in the figure are the etched profiles obtained using a diamond stylus. For a 2-nm-thick SiO_2 layer on Si it was reported [11] that the oxide layer could be etched by Cl_2 induced by an Ar^+ laser at a rate about 80 times lower than the Si–Cl_2 etching. This SiO_2 etch rate may be higher than the real etch rate of a quartz substrate because the underlying Si can absorb visible photons and thus heat the thin oxide layer. In our experiment, the pure SiO_2 wafer does not absorb laser light; therefore, the substrate is neither excited nor heated by the laser beam. Experiments at 514.5 nm with the same laser power produce a much lower etch rate, apparently because the Cl_2 photodissociation yield is much reduced at this longer wavelength. It should be further noted that the diameters of the chemically etched holes [Fig. 3(b)] are much larger than the focal spot, which is estimated to be $\lesssim 5$ μm. This is due primarily to the fact that once the reactive radicals are produced by either multiple-photon dissociation or single-photon photolysis, they can diffuse randomly toward the solid surface to cause chemical etching beyond the focal point region. Because of this, high spatial resolution is generally more difficult to obtain using this method of etching, unless the photon-generated radicals can be confined within a small area just above the solid surfaces by

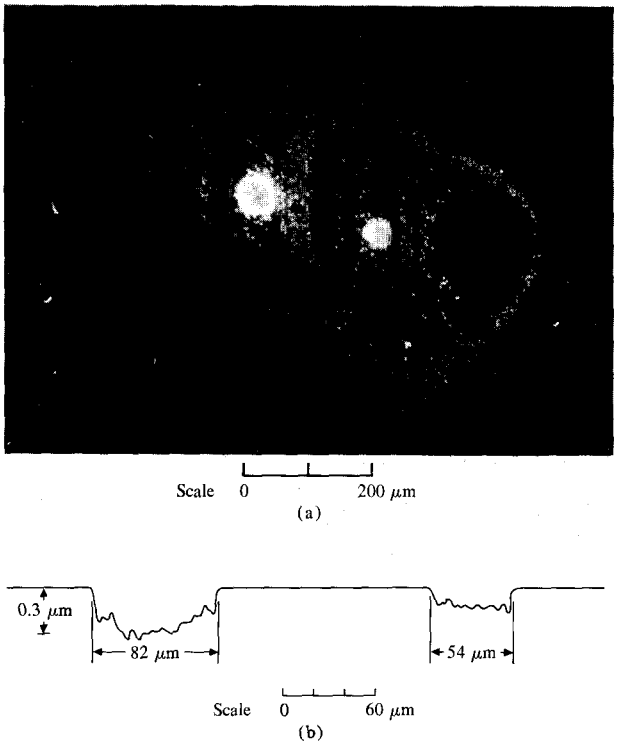

Figure 3 Spots on a quartz wafer etched by Cl_2 gas at 1.3×10^4 Pa photolyzed by a 1.85-W Ar^+ laser at 457.9 nm focused with a 12-mm f.l. lens (a) observed under a 100× microscope magnification and (b) as determined by a diamond stylus. The larger spot on the left side is due to laser-induced etching for 15 min and the smaller spot for 5 min.

some physico-chemical means. It is also interesting to note that although the Cl_2 plasma in the reactive-ion-etching configuration can slowly etch SiO_2 [21], the pure Cl_2 plasma, in which Cl atoms and other species are generated, does not show significant etching of this material [22]. It is possible that under the present experimental condition, the concentration of the Cl atoms in the laser focal point region is higher than that in the Cl_2 plasma. It is also likely that the mechanisms involved in the SiO_2–Cl_2 surface photochemistry are not the same as those involved in the plasma environment.

• *Chemical etching enhanced by laser excitation of solids*

In the preceding sections, laser beams were used to excite gas-phase molecules for the enhancement of surface reactions. These excitation mechanisms are, however, not the necessary conditions for laser-enhanced chemical etching. Surface reactions can be induced by substrate excitation alone. This is illustrated in the CO_2 laser-enhanced interactions of XeF_2 gas with Si, Ta, and Te films. It has been shown that XeF_2 dissociatively chemisorbs on Si [23] and reacts readily with Si at room temperature [24] without any external radiation. The molecules do not absorb ir photons in the CO_2 laser wavelength region covered in the present study. The only effect of laser radiation is lattice excitation and heating; thus, surface fluorine atoms can rearrange to form SiF_4 molecules, which subsequently desorb into the gas phase. As proposed earlier [17], such surface chemical interactions can happen, *e.g.*, via a radiation-induced disproportionation reaction between two "SiF_2"-like surface complexes to form free Si and SiF_4. As a consequence, a localized region of the surface is depleted of fluorine and the sticking coefficient of XeF_2 to Si is increased [4]. Likewise, Ta can react readily with XeF_2 and the surface reaction can be enhanced by CO_2 laser pulses [10].

Molecules of XeF_2 can also chemisorb on Te films, as indicated by substantial decreases in the frequencies of the microbalances when the films are exposed to XeF_2 gas. Unlike Si and Ta, however, Te cannot be continuously etched by the gas without laser radiation. The frequency response of the Te microbalance under a XeF, partial pressure of $\lesssim 1.3$ Pa and irradiated with CO_2 laser pulses is shown in Fig. 4. For $\Delta f = 360$ Hz/8 pulses, $EY \approx 1.4 \times 10^{15}$ Te atoms/pulse. Among the gas–solid systems examined in the present study, the Te–XeF_2 result shows the largest enhancement of etching yield produced by the CO_2 laser (see Table 1). Because the excitation of gaseous XeF_2 molecules is not necesary for the enhancement, it is clear that as long as the solid substrates absorb the laser light, the surface chemistry of the gas–solid systems can be promoted just as well by lasers in the uv-visible regions.

Summary

The behavior of the laser-enhanced chemical etching of Si and Ta with SF_6 has been investigated in some detail. We have also studied the Si, Ta, and Te interactions with XeF_2 in conjunction with a pulsed CO_2 laser, and the SiO_2–Cl_2 reaction with a cw Ar^+ laser. The enhanced surface reactions could be divided into three categories: those activated by vibrational excitation of etchant molecules, those due to radicals generated by photodissociation, and those induced by laser excitation of solid substrates. The presence of laser radiation at gas–solid interfaces can promote one or more of these photochemical processes. To achieve the desirable etch rates and spatial resolution for a given solid material of interest, one must consider the choices of gaseous chemicals, suitable lasers (*i.e.*, wavelength, intensity, pulse width, pulse rate, and laser mode), in addition to other parameters such as gas pressure and substrate temperature. Compared to conventional plasma etching, laser chemical etching is still rather slow in many cases, particularly in terms of the total amount of material to be processed in a given period of time. Nevertheless, the laser method has many distinct advantages, and it can be particularly useful for personalized material processing. Direct maskless etching can be

achieved by this technique with good directionality, selectivity, and the essential absence of many problems usually associated with plasma etching, such as loading effects and the dependence of etch rate on the reactor and vacuum system used. The glow discharge in a plasma environment also generates more reactive and hazardous by-products. The present experiment shows that the mechanisms involved in plasma etching and laser-enhanced chemical etching can be quite different. Furthermore, for solid substrates that absorb focused laser photons, a localized spot on a substrate can be excited and heated to a rather high temperature. At such temperatures, the surface chemistry, including the volatility of etching products, can be very different from the relatively low-temperature reactions normally encountered in a plasma environment. Because of the substantial differences in etching mechanisms between plasma chemistry and laser chemical etching, it is very possible that there may be instances, such as the SiO_2–Cl_2 system, in which solid materials can be etched efficiently by the laser method but only ineffectively by the plasma technique. For applications such as localized repair of microcircuits and localized formation of spots or trenches, the laser approach can be especially valuable. It is therefore reasonable to expect that more studies will be attempted in the future and more applications to materials processing will be found using laser techniques.

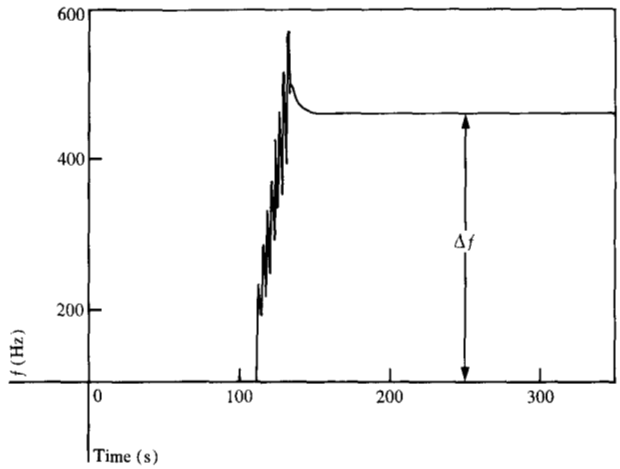

Figure 4 The frequency response of the Te microbalance excited by CO_2 laser pulses at 975.9 cm^{-1} with I = 1 J/cm^2, normal incidence, in XeF_2 gas at 1.3 Pa; Δf = 360 Hz/8 pulses.

Table 1 Pulsed CO_2 laser-enhanced chemical etching of Si, Ta, and Te films by XeF_2 gas measured with quartz-crystal microbalances; CO_2 laser at 942.4 cm^{-1}, I = 1.0 J/cm^2, normal incidence. The XeF_2 partial pressure is $\lesssim$0.013 Pa for Si and Ta systems, and $\lesssim$1.3 Pa for the Te systems (see text). The experimental errors are about ±20%.

Material	Δf (Hz/laser pulse)	*Enhanced etching yield* (nm/laser pulse)
Si	1.1	0.06
Ta	4.9	0.04
Te	48.0	1.01

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