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Laser-induced desorption from silicon (111) surfaces with adsorbed chlorine atoms

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Abstract. We have studied the initial stage of the laser-induced reaction of silicon (111)- 7×7 surfaces with adsorbed chlorine atoms in ultrahigh vacuum, by measuring the species (SiCl_x) desorbing from the surfaces. In particular, our studies have focused on photo-chemical etching without laser-induced thermal heating. We found that the primary species desorbing from Cl-saturated Si(111) surfaces is the SiCl_2 molecule and that the desorption efficiency with 2.3, 3.5 and 4.7 eV photons is significantly enhanced with respect to that for 1.2 eV photons. The results of previous STM studies are discussed and a possible mechanism for the photo-chemical etching is proposed.

1. Introduction

Laser-induced processes on Si surfaces with adsorbed chlorine atoms have attracted a great deal of attention in both applied and pure physics. Much work [1–11] has been done on applications to device fabrication, especially photo-chemical etching, and on the understanding of fundamental electronic processes. However, these experiments treated complicated processes due to ambient gas or to thermal effects induced by high-power laser irradiation. Recently, chlorinated and gently irradiated surfaces have been studied in ultrahigh vacuum (UHV) with a scanning tunnelling microscope (STM), and the desorption sites due to electronic excitation have been clarified on an atomic scale [12–14]. Thus, for understanding photo-stimulated atomic processes at the surfaces, significant information will be given by a combination of laser-induced desorption (LID) measurements and STM observations on resulting surfaces under both UHV and non-thermal conditions.

For the clean Si(111)- 7×7 -reconstructed surface structure, the dimer–adatom–stacking-fault (DAS) model [15] is widely accepted. A schema of the 7×7 DAS model is shown in the left-hand half in figure 1. A unit cell in the DAS model includes 12 adatoms (solid), 42 rest atoms (grey), 9 dimers, and 1 corner hole. Adatom positions in the DAS model correspond to atomic protrusions in STM topographs with a sample bias voltage V_s higher than about +0.5 V [16, 17, 18]. These Si-adatom protrusions in STM images are attributed to a Si-adatom empty state located about 0.5 eV above the Fermi level E_F .

Using an STM and scanning tunnelling spectroscopy (STS), Villarrubia and Boland [19, 20] have shown that a Cl atom sticks on top of a Si adatom, forming a silicon-adatom monochloride SiCl (labelled 'M' in figure 1) [21, 22], on the Si(111)- 7×7 surface at low chlorine coverage. Their STS measurements indicate that empty states on a Cl-reacted Si adatom are at levels higher than $E_F + 3$ eV. This result demonstrates that adatom

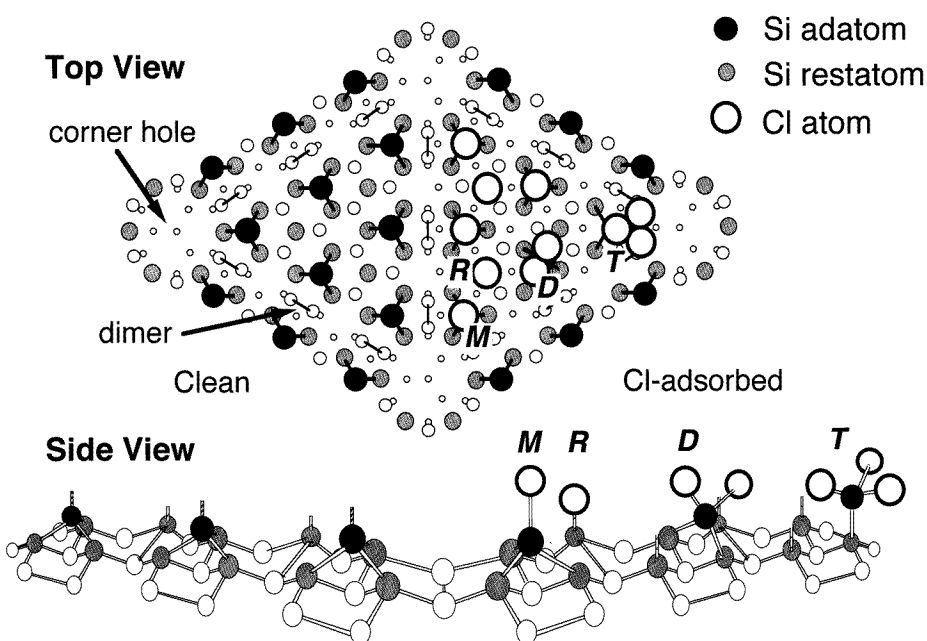


Figure 1. Schematic views of the Si(111)- 7×7 DAS structure, clean (the left-hand half-unit) and Cl-adsorbed (the right-hand half-unit). A unit cell of the 7×7 structure is surrounded by 4 corner holes and 12 dimers, and includes 12 Si adatoms and 42 Si rest atoms. The symbols M, D, T, and R denote silicon-adsatom mono-, di-, and trichlorides, and silicon-rest-atom monochloride, respectively. The arrangement of these silicon chlorides is arbitrary. The side view shows a front area of the top view.

monochloride sites can be distinguished from bare Si-adsatom sites by measuring the V_s -dependence in STM topographs.

At high chlorine coverage, x-ray photoemission spectroscopy (XPS) studies have demonstrated that chlorine molecules are adsorbed dissociatively on the Si(111)- 7×7 surfaces, forming SiCl, SiCl₂, and SiCl₃ [21–24]. A structural model for SiCl_x ($x = 1–3$) was proposed on the basis of the measured positions of the SiCl_x species with respect to the atomic lattice of the substrate in the STM topograph [19, 20]. The proposed model of SiCl_x on the 7×7 DAS surface is described in the right-hand half of figure 1, their arrangement being however arbitrary. A silicon-adsatom dichloride SiCl₂ (labelled ‘D’) and an adsatom trichloride SiCl₃ (labelled ‘T’) are formed by Cl atoms which break the bonds between the Si adsatoms and the Si rest atoms, and bond to them. Consequently, the Si-adsatom polychlorides relax from the original positions of the bare Si adsatoms, as seen in figure 1. In addition, Cl atoms terminate Si rest atoms forming Si-rest-atom monochlorides (labelled ‘R’) since the density of SiCl species on saturated Cl/Si(111) surfaces is more than that of the bare Si adsatoms on clean surfaces [21–24].

When chlorine-saturated surfaces are heated up to about 400–500 °C, it is primarily a SiCl₂ molecule that is desorbed [22, 25]. After the desorption, only the SiCl species is observed and the SiCl₂ and the SiCl₃ species disappear in the XPS measurement [21]. The ordered structure of the annealed surface is found to be the same as that of the rest atoms of the 7×7 DAS model in the STM images [19, 20, 12]. Irregular islands of silicon

chlorides are also found on the annealed surfaces. The surface species was identified with the Si-rest-atom monochloride ('R') by STS [19, 20]. These results indicate that the adatom polychlorides on the chlorine-saturated Si(111) surfaces are bound weakly to the substrate, and diffuse to form islands and/or are desorbed as SiCl₂ molecules by annealing at these temperatures. On the other hand, the rest-atom monochlorides are stable against the annealing.

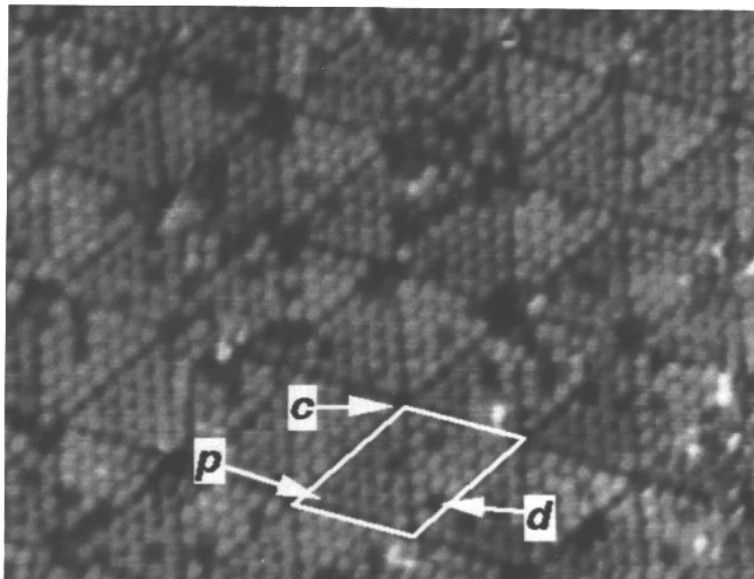


Figure 2. An STM image obtained after irradiation with laser pulses of a chlorine-saturated Si(111) surface [14]. The photon energy is 4.7 eV and the sample bias voltage is +3.0 V. A white rhombic indicator shows a unit cell surrounded by corner holes ('c') and dimers. Bright spherical protrusions ('p') on an atomic scale are 7×7 ordered as the Si rest atoms of the DAS model. A number of depressions ('d') are found on the surface especially around corner holes and dimers.

Effects of laser irradiation in UHV on Si(111) surfaces with adsorbed chlorine atoms have been studied with STM [12, 13, 14]. Figure 2, due to Shudo *et al* [14], is an STM topograph with $V_s = +3.0$ V of a Cl-saturated Si(111) surface after irradiation with pulsed laser light having a photon energy of 4.7 eV. The resulting surface was flat, showing rest-atom 7×7 -ordered protrusions (labelled 'p' in figure 2) with a number of depressions (labelled 'd') without any islands, in contrast to what is seen on Cl-saturated Si(111) surfaces after annealing. Since a laser fluence of about 10 mJ cm^{-2} in the experiments does not cause any thermal heating [10], Shudo *et al* considered that the adatom polychlorides did not diffuse and were desorbed out by electronic processes. They assigned the rest-atom 7×7 -ordered protrusions ('p') to the silicon-rest-atom monochlorides, and attributed the depressions ('d') either to point defects or to atoms with empty states much higher than $E_F + 3.0$ eV. They demonstrated that both the adatom monochloride and the rest-atom monochloride are stable against laser stimulation, while the adatom polychlorides are unstable. The photon energy dependence of the laser stimulation on Cl/Si(111) surfaces in the STM images shows that excitation of 2.3 and 1.2 eV photons can induce the desorption of adatom polychlorides. They found that the density of the remaining adatom polychlorides

increases with decreasing photon energy.

The LID from silicon surfaces in ambient Cl_2 gas has been studied by several authors [4, 9, 10, 11]. Neutral SiCl and SiCl_2 molecules are major desorption products. However, the initial stage of the desorption has not been studied so far because almost all LID experiments have been carried out in ambient Cl_2 gas. The etching depth was large enough to be measured by a mechanical stylus.

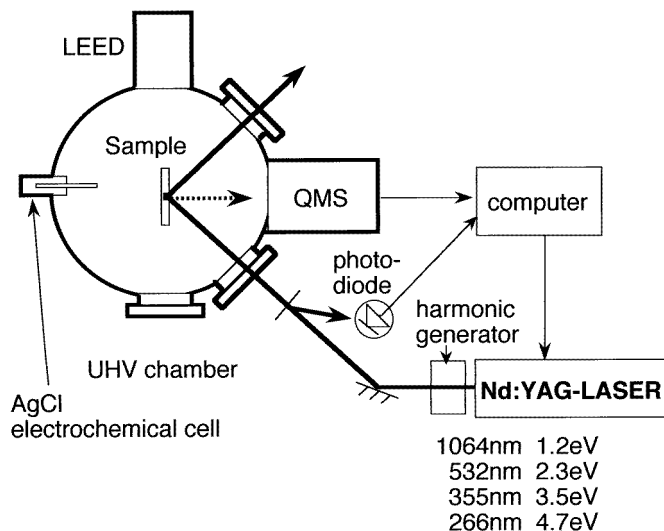


Figure 3. The experimental set-up for laser-induced desorption measurements.

In this paper, we present results of LID measurements for the photo-chemical etching of the topmost surface monolayer of $\text{Si}(111)-7 \times 7$ surfaces with adsorbed chlorine atoms in UHV under the condition that the laser fluences are low enough for preventing thermal heating. Using the results from the LID and the previous STM [14] measurements, we discuss a mechanism for the very initial stage of the etching on an atomic scale.

2. Experimental details

As shown in figure 3, we used a UHV chamber with low-energy electron diffraction (LEED) optics (Varian 981-0127) and a quadrupole mass spectrometer (QMS) (ULVAC MSQ-150). The base pressure of the UHV chamber was lower than 1×10^{-8} Pa. A sample of size $10 \times 20 \times 0.3 \text{ mm}^3$ was cut from the same $\text{Si}(111)$ wafer (P-doped, $2 \Omega \text{ cm}$, n-type) as used in the previous STM observations [14]. A clean $\text{Si}(111)-7 \times 7$ surface was obtained by direct-current (DC) heating to $1200 \text{ }^\circ\text{C}$ in UHV. Chlorine gas was dosed to the surface up to several tens of langmuirs with a AgCl electrochemical cell [26]. This dose is known to be enough for the saturation of Cl on the surface [27].

Multiple-harmonic s-polarized light from a pulsed Nd:YAG laser (Spectra Physics GCR-11, 10 Hz repetition frequency) was employed to stimulate the $\text{Si}(111)$ surfaces at the saturation Cl coverage. The incidence angle of the laser was 50° and the spot diameter was about 3 mm. The photon energies (and pulse durations) of the fundamental, the second, the third and the fourth harmonics are 1.2 eV (8.5 ns), 2.3 (6.5), 3.5 (5.5) and 4.7 (4.5),

respectively. The laser power for each pulse was monitored by a photodiode detector calibrated with a power meter (Nihon Kagaku, PM-322).

Laser-induced desorbed species were detected by the QMS, whose electron impact ionizer is located in front of the sample 3 cm away. We tuned m/e to 63, 98, and 133, corresponding to SiCl^+ , SiCl_2^+ , and SiCl_3^+ , respectively. Signals from the secondary-electron-multiplier output of the QMS were stored in a microcomputer through an analogue-to-digital converter. We evaluated the minimum yield detectable per laser pulse to be of the order of 10^{-4} monolayer (ML). This value was estimated from the thermal desorption yield of SiCl_x from the Cl-saturated Si(111) surfaces with the same apparatus by DC heating.

3. Results

We detected SiCl^+ and SiCl_2^+ when chlorine-saturated Si(111) surfaces were irradiated above certain threshold laser fluences which depend on the photon energy. The measured threshold fluence for SiCl^+ detection at each photon energy is the same as that for SiCl_2^+ detection within the accuracy of the measurement, 20%, while we could not detect SiCl_3^+ below 1 J cm^{-2} for all photon energies. The same threshold fluence signifies that the same origin results in the SiCl^+ and the SiCl_2^+ detection. The detectable threshold fluences were 0.02, 0.03, 0.04 and 1 J cm^{-2} for photon energies of 4.7, 3.5, 2.3 and 1.2 eV, respectively, in our QMS sensitivity (figure 4). For a 1.2 eV photon, obvious laser ablation [10, 28] was observed at fluences just above 1 J cm^{-2} .

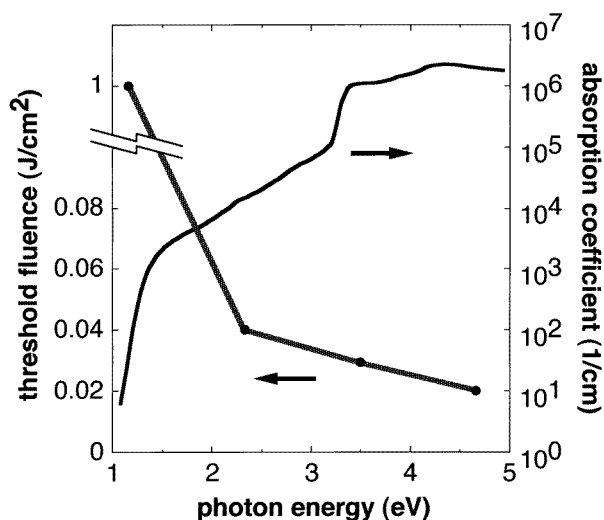


Figure 4. The photon energy dependence of the threshold fluence for the detection of SiCl_2 desorption from Cl-saturated Si(111) surfaces. The absorption coefficient of bulk silicon [33, 34] is also drawn as a function of photon energy.

No SiCl_x^+ were detected above the threshold fluences when the ionizer was switched off. Therefore, the observed molecules were not desorbed ion molecules but ionized or ionized-cracked molecules made from desorbed neutrals.

When Cl-saturated Si(111) surfaces were heated up to $500 \text{ }^\circ\text{C}$, we also detected SiCl^+ and SiCl_2^+ made from neutrals. The yield ratio of SiCl^+ to SiCl_2^+ was about 3. Since

the dominant thermal desorption species at 420 °C is SiCl₂ [22, 25], the cracking of SiCl₂ neutrals in the ionizer increases the ratio to 3 in our apparatus.

We measured total yields of SiCl⁺ and SiCl₂⁺ by laser stimulation for several tens of laser pulses at 10–30% above the threshold fluences. The yield ratio of SiCl⁺ to SiCl₂⁺ was about 3, and was independent of the photon energies within 20% accuracy. Because the ratio is the same as in thermal desorption, we assigned primary LID species to a SiCl₂ molecule.

4. Discussion

In order to estimate the number of SiCl₂ desorbed by a laser-induced thermal heating, we calculated *t*- (time) and *z*- (distance from the surface) dependent silicon temperatures $T(t, z)$ using a simple equation [29, 30]: $c(\partial T/\partial t) - \kappa(\partial^2 T/\partial z^2) = \alpha I_0 \exp(-\alpha z)$. Here *c* is the specific heat of silicon, κ is the thermal conductivity, α is the absorption coefficient, and I_0 is a laser intensity. The highest temperature calculated is about 700 °C and continues for several ns in the case of a fluence of 0.1 J cm⁻² with a photon energy of 4.7 eV. Gupta *et al* [25] reported that the thermal desorption rate $d\theta/dt$ of SiCl₂ is described as $\theta^2 \nu_d \exp(-E_d/RT)$, where the desorption pre-exponential ν_d is 3.2 cm² s⁻¹, the desorption activation energy E_d is 67 kcal mol⁻¹, and the chloride coverage θ is 5×10^{14} cm⁻² assuming a saturation dose of chlorine atoms. Thus, we estimate the maximum number of molecules desorbed thermally by laser irradiation to be about 10⁻⁷ ML per pulse, which is much smaller than our detection sensitivity. Furthermore it was reported that non-thermal reaction occurs at fluences below about 150 mJ cm⁻² [10]. Actually, our threshold fluences except for 1.2 eV photons are in this low-power regime. Therefore we conclude that electronic processes dominate the thermal effect in the desorption at fluences below several tens of mJ cm⁻².

The following three-step scenario is widely used to explain electronic processes leading to desorption [31, 32]. Step 1: photon or electron stimulation induces electronic transitions at the surface or in the bulk. Step 2: electronic energy from the initial excitation localizes in or around the desorption sites for a long enough time. Step 3: bond instability at the site occurs by virtue of the localized excitation, resulting in desorption.

We consider that this scenario is also applicable to the LID phenomena on the Cl/Si(111) surface. There are two possible transition processes for step 1: a transition in the bulk or a transition involving chlorine-induced states at the surface. We found that there is a drastic difference in the threshold laser fluence for photon energies between 1.2 eV and 2.3 eV. On the other hand, the thresholds are not so different for photon energies above 2.3 eV as shown in figure 4. In this figure the absorption coefficient of bulk silicon [33, 34] is also plotted as a function of photon energy. We note that it increases rapidly from 1.2 eV to 1.5 eV. These facts suggest that the desorption is induced by electronic excitation in the bulk.

Similar mechanisms have been discussed for NO/Si(111) surfaces [35] and Cl/Si(111) surfaces [9–11]. In the case of NO/Si(111)-7 × 7 surfaces, a photon energy dependence of the LID yield indicates that the desorption is induced by bulk excitation; a drastic increase of the NO desorption yield was observed with increasing photon energy from 3.0 to 3.3 eV. The range of the photon energy is different from that in our work. Rhodin *et al* [10, 11] have reported that the LID yield from Cl/Si(111) in ambient Cl₂ gas is enhanced for heavily doped specimens of both n and p type. They have suggested that photo-generated holes or electrons in the bulk are accelerated to the surface by band bending.

After 1.2 eV irradiation at 10 mJ cm⁻², slight changes of a partially chlorinated surface

were observed in the STM image [14], although we could not detect any SiCl_x ($x = 1-3$) species below 1 J cm^{-2} in our LID measurements. The STM result suggests the desorption of polychlorides even with 1.2 eV photons. The disagreement between the results obtained from LID and those obtained by STM may be attributed to the sensitivity of the former being lower than that of the latter; the LID needs a desorption yield of more than 10^{-4} ML for each pulse, while the STM can detect consequences of the desorption if the desorption sites are at least of the order of 10^{-3} ML after 6000 pulses.

Desorption by 1.2 eV photons suggests electronic transitions of chlorine-induced surface states in the band gap of the bulk silicon as step 1 in the LID scenario. That is, electronic transitions occur not only in the bulk but also at the surface. These different transition paths may explain the photon energy dependence of the LID yield reported by Qin *et al* [9]. They demonstrated that the LID yield of SiCl_2 from Si(111) surfaces in ambient Cl_2 gas for 1.2 eV photons is smaller than that for photons of energy above 2.3 eV, and that the yield is a linear function of the fluence for the latter while it is non-linear with higher order for the former.

Steps 2 and 3 in the LID scenario suggest that excited holes or electrons around the silicon-adatom polychlorides on the surface destabilize the bonding of the polychlorides to the substrate, but not the bonding of the silicon-adatom monochlorides. This agrees with the qualitative arguments that a monochloride is strongly bound to the substrate with three bonds, and that di- and trichlorides are weakly bound with two and one bonds.

According to semiempirical calculations for $\text{Si}_{18}\text{H}_{27}\text{Cl}_n$ ($n = 0-7$) clusters which are models of chlorine-adsorbed Si(111)- 7×7 surfaces, the energies of decomposition of SiCl from a $\text{Si}_{18}\text{H}_{27}\text{Cl}$ cluster, SiCl_2 from $\text{Si}_{18}\text{H}_{27}\text{Cl}_4$, and SiCl_3 from $\text{Si}_{18}\text{H}_{27}\text{Cl}_5$ are estimated to be 4.52, 2.40, and 0.88 eV, respectively [36]. If we assume that these values are close to the binding energies for silicon-adatom chlorides on Cl/Si(111) surfaces, the bonds of the silicon polychlorides can be broken energetically by the localization of one or more excited holes or electrons which are generated by photons with energies of a few eV.

We observed the LID species of SiCl_2 primarily rather than that of SiCl_3 at Cl-saturated Si(111) surfaces. This suggests that a silicon-adatom trichloride is desorbed dissociatively as a SiCl_2 molecule, and a chlorine atom is left on the substrate, forming a silicon-rest-atom monochloride. We can estimate the energy gain of dissociative desorption of SiCl_3 as SiCl_2 from the $\text{Si}_{18}\text{H}_{27}\text{Cl}_5$ cluster in the following way. The decomposition energy E_1 of SiCl_3 from this highly chlorinated cluster is 0.88 eV. The experimental value of the energy of fragmentation E_2 of a SiCl_3 molecule to a SiCl_2 molecule and a Cl atom is 2.86 eV [37, 38]. The above calculations [36] also show 3.61 eV of the energy gain E_3 when a Cl atom is put on a $\text{Si}_{17}\text{H}_{27}\text{Cl}_2$ cluster, corresponding to forming a silicon-rest-atom monochloride structure. Thus we obtain the dissociative desorption energy of SiCl_3 from the $\text{Si}_{18}\text{H}_{27}\text{Cl}_5$ cluster: $E_1 + E_2 - E_3 = 0.13$ eV, which is smaller than E_1 .

On the LID-resulting surface (figure 2), we notice that most of the depressions ('d') are next to the dimers or the corner holes rather than at the other sites. Since the rest-atom monochlorides are stable against irradiation, we can ascribe the depressions around the dimers or the corner holes to point defects made as consequences of the desorption of the rest-atom polychlorides. The existence of the rest-atom polychlorides was reported from XPS measurements [23]; the density of SiCl_2 and SiCl_3 on the Cl-saturated Si(111) surface is higher than that of bare Si adatoms on a clean 7×7 surface. It seems to be easy for chlorine atoms to break the bonds between the substrate and the rest atoms adjacent to the dimers or the corner holes, as seen in figure 1.

In summary, photo-chemical etching of silicon (111) surfaces with adsorbed chlorine atoms progresses in the following way. First, incident photons generate excited holes or

electrons mainly in the bulk. Second, the excited carriers are diffused or accelerated to the surface, and are localized at desorption sites for a long enough time. At the initial stage of the etching, silicon-adatom polychlorides and rest-atom polychlorides are destabilized by the localization of excited carriers at these sites. Finally, these silicon polychlorides are desorbed as SiCl_2 molecules primarily, and the resulting surface consists of silicon-rest-atom monochlorides with point defects.

5. Conclusions

We have studied laser-induced desorption from chlorine-saturated Si(111) surfaces in UHV with fluences low enough to prevent thermal effects. The primary desorption species is a SiCl_2 molecule and the detection threshold fluences with photon energies between 4.7 eV and 2.3 eV are over ten times smaller than that for 1.2 eV. The drastic increase in the desorption efficiency with photons of energies above 2.3 eV implies that the desorption is dominantly induced by photo-generated bulk carriers. The qualitative change of STM images [14] after 1.2 eV irradiation suggests a slight contribution of an electronic transition involving Cl-induced surface states to the desorption. The STM study indicates that silicon-adatom polychlorides and silicon-rest-atom polychlorides are unstable to laser irradiation, while silicon-adatom monochlorides and silicon-rest-atom monochlorides are stable.

The desorption of the adatom polychlorides results in silicon-rest-atom monochlorides forming 7×7 -ordered surfaces. On the other hand, the desorption of the rest-atom polychlorides results in point defects.

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