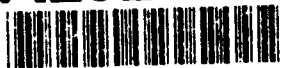


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Comparison of Cl_2 and HCl Adsorption on $\text{Si}(100)-(2 \times 1)$

by

Q.Gao, C.C. Cheng, P.J. Chen, W.J. Choyke and J.T. Yates, Jr.

Submitted to:

2nd International Symposium
on Atomic Layer, Epitaxy
Session E-12

Surface Science Center
Department of Chemistry
University of Pittsburgh
Pittsburgh, PA 15260

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Abstract

The chemisorption and reaction of Cl_2 and HCl on $\text{Si}(100)-(2 \times 1)$ have been studied using a variety of measurement methods. At 100K, both Cl_2 and HCl dissociatively chemisorb on the dangling bonds of $\text{Si}(100)$. At saturation coverage, the surface concentration of Cl is about one Cl atom per Si atom while HCl can produce a Cl coverage only one fourth of that achieved with Cl_2 . This is the first report of a self-site-blocking effect in adsorption on a semiconductor surface. HREEL spectra indicate that both Cl_2 and HCl adsorption at 100K give a monochloride surface species with a Si-Cl stretching frequency of $\sim 550\text{--}600\text{ cm}^{-1}$. Digital ESDIAD measurements reveal that the Si-Cl bond angle for the monochloride from both Cl_2 and HCl adsorption is oriented on the vertical plane containing the Si-Si dimer bond and is inclined from

the surface normal. The etching products of Si(100) by Cl_2 at elevated temperatures are SiCl_2 at $\sim 800\text{K}$ and a small amount of SiCl_4 at $\sim 500\text{K}$. For HCl , the only observed etching product is SiCl_2 .

1. Introduction

It is important to understand the basic chemistry of chlorine chemisorption and reaction on semiconductor surfaces, since Cl-containing molecules, such as SiH_2Cl_2 [1] and GaCl_3 [2], have been used as precursor molecules for atomic layer epitaxy processes, and Cl_2 molecules have been used in the ion [3-5] or photon assisted etching [6-8] process.

In this report, the saturation coverage and vibrational spectrum of Cl(a) derived from Cl_2 and HCl adsorption are compared on Si(100). In addition, Cl^+ ESDIAD patterns from Si-Cl bonds on Si(100) are reported, giving information about the Si-Cl bond direction.

2. Experimental

Experiments were carried out in two UHV chambers. The first one was equipped with a digital ESDIAD/LEED (electron stimulated desorption ion angular distribution/low energy electron diffraction) apparatus, an Auger electron spectroscopy (AES), a quadrupole mass spectrometer (QMS) for line-of-sight temperature programmed desorption (TPD), and an additional QMS for ion mass analysis in ESD. The second UHV chamber housed a high resolution electron energy loss spectrometer (HREELS), LEED, AES and a QMS for TPD. The primary beam energy used for HREELS study was 4.2 eV

and the full width at half maximum (FWHM) of the elastic beam was about 65 cm^{-1} . Flux-calibrated microcapillary-collimated gas dosers [9,10] were used in both chambers for control of the Cl_2 or HCl exposure. The $\text{Si}(100)$ single crystal was cleaned by Ar^+ sputtering and subsequent annealing at 1173K . The crystal temperature was measured by a chromel-constantan thermocouple enclosed in a Ta-foil envelope which was inserted into a slot on the crystal edge [9].

3. Results and Discussion

3.1. Adsorption of Cl_2 and HCl

The saturation coverage of the chlorine layer derived from the adsorption of Cl_2 and HCl was measured. Figure 1 is an Auger measurement of the development of the $\text{Cl}(a)$ coverage using the two molecules as a function of exposure. It is noticed that at about 3×10^{14} molecules/ cm^2 exposure, both the Cl_2 and HCl adsorption rate has dramatically decreased. The reduction of the adsorption rate for Cl_2 is due to the lack of available surface dangling bonds since all of them are nearly used up for the chemisorption [11]. The perfect $\text{Si}(100)-(2 \times 1)$ surface exposes 6.8×10^{14} dangling bonds/ cm^2 . Thus, at a Cl_2 exposure near $\sim 3 \times 10^{14} \text{ Cl}_2/\text{cm}^2$, saturation of the dangling bonds is expected to occur (near the break point in the curve) assuming a sticking

probability of unity in the initial adsorption region. Beyond the break point at $\sim 3 \times 10^{14}$ Cl_2/cm^2 , the coverage continues to rise slowly as the Cl_2 exposure is increased. For the same exposure ($\sim 3 \times 10^{14}$ molecules/ cm^2), the saturation effect is also observed for HCl adsorption. The surface Cl coverage from HCl adsorption is only about one-fourth of that achieved by Cl_2 adsorption, as obtained from the Cl/Si AES intensity ratio in Figure 1. The Cl_2 molecules are composed of two Cl atoms which give a Cl coverage of nearly one Cl atom per dangling bond at the break point. In comparison, HCl is composed of one Cl atom and one H atom. If the surface dangling bonds were all occupied by H(a) and Cl(a) from HCl adsorption, a Cl surface coverage of about 0.5 would be expected. However, at the break point only about one fourth of the surface dangling bonds are occupied by Cl from HCl adsorption at 100K. The surface has one half of the dangling bonds which are not accessible for HCl adsorption. Furthermore, after a saturation exposure of HCl is achieved, exposure of this prepared surface to Cl_2 leads to increased Cl coverages as measured by AES, indicating that the dangling bonds inaccessible for HCl adsorption are active for Cl_2 adsorption. Thus, the HCl adsorption on Si(100) is a self-site-blocking process. To our knowledge, this is the first report of such a self-site-blocking adsorption process on a semiconductor surface. The immediate conclusion from this result is that HCl will be a less effective etchant than

Cl₂ due to its limited surface coverage on Si(100). The nature of this self-sit-blocking adsorption phenomena is not well understood at present.

3.2. Vibrational Studies of Cl₂ and HCl Adsorption

Deeper insight into the chlorine surface chemistry can be obtained from the vibrational characterization of the surface species formed during the chemisorption and reaction. Figure 2 shows the vibrational spectra after Cl₂ and HCl adsorption on Si(100)-(2x1) at 100K. It is noticed that only one strong Si-Cl stretching mode, $\nu(\text{SiCl})$, is observed at $\sim 600 \text{ cm}^{-1}$ and at $\sim 550 \text{ cm}^{-1}$ for Cl₂ and HCl adsorption, respectively [12]. For HCl adsorption, an additional vibrational feature is observed at $\sim 2120 \text{ cm}^{-1}$ (figure 2b) which is due to the SiH stretching mode [13]. The lack of a HCl stretching mode near $\sim 3000 \text{ cm}^{-1}$ [14] and the appearance of $\nu(\text{SiCl})$ and $\nu(\text{SiH})$ modes indicate that HCl is dissociated upon adsorption at 100K.

The vibrational spectra (Figure 2) indicate that higher surface chloride species, such as SiCl₂(a), SiCl₃(a) or SiCl₄(a), are not observed following either Cl₂ or HCl adsorption at 100K. These chlorides should exhibit an asymmetric stretching mode [$\nu_a(\text{SiCl}) \sim 533-617 \text{ cm}^{-1}$], and a symmetric stretching mode [$\nu_s(\text{SiCl}) \sim 376-465 \text{ cm}^{-1}$]; in addition a bending vibrational mode should be present [$\delta(\text{Si-Cl}) \sim 164-261 \text{ cm}^{-1}$] [12a-e]. There is no prominent

evidence for these modes in the spectra observed. This is consistent with the dissociative adsorption of HCl and Cl₂ forming surface monochloride species. For Cl₂ adsorption at 100K, the mode at 295 cm⁻¹ has been assigned to a Si₂Cl stretching mode for a bridge-bonded Cl species, a minority species [15].

3.3. ESDIAD Studies of Si-Cl Bonding on Si(100)

The ESDIAD method has been employed to study the bonding of Cl(a) to Si(100) using both Cl₂ and HCl as adsorbates. In this method, the adsorbed layer is bombarded by electrons ($V_e=120$ eV in this case) and the angular distribution of Cl⁺ ions is detected by a digital technique. The ejection angle of the Cl⁺ ions is determined primarily by the orientation of the Si-Cl bond being broken [16-18] in the electronic excitation. This angle is modified by final state effects involving the Cl⁺ interaction with its image charge as well as by Cl⁺ reneutralization near the surface [19-20].

The surface monochloride species produced from HCl or Cl₂ adsorption give four off-normal Cl⁺ emission beams observed from the ESDIAD measurements, shown in Figure 3. The Cl⁺ ions are ejected in planes perpendicular to the crystal surface but parallel to the surface Si-Si dimer bond axes, as confirmed from the comparison of the ESDIAD pattern with the (2x1) LEED patterns of the substrate

surface. Slight misalignment of the Si single crystal from the (100) direction results in a stepped surface with Si-Si dimer rows oriented alternately in orthogonal directions, forming (1x2) and (2x1) domains of reconstructed surface. The inclined Cl bonds of the surface monochloride species from both types of domains produce the four off-normal Cl^+ ion beams. A single domain surface would give two off-normal Cl^+ ion beams with the opposite polar angle with respect to the surface normal.

3.4. Thermal Desorption Studies

The thermally activated etching reaction was investigated by TPD measurements. The results (Figure 4) indicate that Cl(a) from both Cl_2 and HCl chemisorption etches the Si(100) surface, producing $\text{SiCl}_2(\text{g})$ with a desorption peak temperature at ~820K for the $\text{Cl}_2/\text{Si}(100)$ system and at 840K for the HCl/Si(100) system. A small amount of SiCl_4 desorption (monitored by its major mass spectrometer cracking product, SiCl_3^+ , $m/e=133$ amu) is also observed for the $\text{Cl}_2/\text{Si}(100)$ system at monolayer or higher exposures. This is consistent with the previous $\text{Cl}_2/\text{Si}(100)$ TPD results [6, 21]. These measurements indicate that SiCl_2 is the main etching product for both Cl_2 and HCl adsorption, and that the etching process involving SiCl_2 occurs near 840K. Species such as SiH_4 (monitoring $m/e=30$ amu) or SiH_3Cl (monitoring $m/e=31$ amu), SiH_2Cl_2 (monitoring

m/e=99 amu) and SiHCl_3 (monitoring m/e=133 amu) were not detected in desorption, showing that the surface hydrogen atom was not involved in the etching process when HCl was employed.

An effort was made for the $\text{Cl}_2/\text{Si}(100)$ system to observe by HREELS whether the $\text{SiCl}_2(\text{a})$ species could be detected by raising the crystal temperature to $\sim 850\text{K}$, followed by cooling to 100K . The HREEL spectrum remained essentially the same, exhibiting a single Si-Cl stretching mode of the surface monochloride shown in Figure 2a with a reduced intensity and a slight reduced frequency (to about 570 cm^{-1}) due to the reduction of surface chlorine coverage. Thus, $\text{SiCl}_2(\text{a})$ formation is probably the rate determining step for $\text{SiCl}_2(\text{g})$ liberation.

4. Conclusions

The following conclusions may be made from these studies of Cl_2 and HCl chemisorption on $\text{Si}(100)-(2\times 1)$.

- (1). Dissociative adsorption of both Cl_2 and HCl occurs on $\text{Si}(100)-(2\times 1)$ at 100K . The vibrational spectra of the species produced from Cl_2 and HCl adsorption indicate that Si-Cl bonds are formed in both cases. For HCl chemisorption, the Si-H bond is also observed.
- (2). For Cl_2 adsorption, about one Cl atom/Si dangling bond is produced at saturation

coverage. In addition, the saturation coverage of chlorine from Cl_2 adsorption is ~4 times greater than that from HCl adsorption. This result implies that HCl adsorption involves an adsorption site exclusion process, where neither H(a) or Cl(a) species can populate one-half of the dangling bond sites exposed on Si(100)-(2x1) .

- (3). ESDIAD studies indicate that the Si-Cl bonds are inclined away from the surface normal direction along perpendicular planes containing the Si-Si dimer bonds.
- (4). SiCl_2 is the main etching product observed from thermal desorption studies of Cl_2 and HCl on Si(100) . It desorbs near 800K in both cases. HREELS studies indicate that $\text{SiCl}_2(\text{a})$ is not produced in measurable amount on heating the surface. This indicates that the rate determining step for $\text{SiCl}_2(\text{g})$ desorption is the conversion of SiCl(a) species to $\text{SiCl}_2(\text{a})$ species which then desorb.

5. Acknowledgments

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11. Cross calibrations of surface Cl coverage from Cl₂ adsorption on Si(100) with the saturation coverage of monohydride from atomic hydrogen adsorption indicate

that the surface Cl coverage is about 0.9 at the break point of the Cl₂ AES uptake curve.

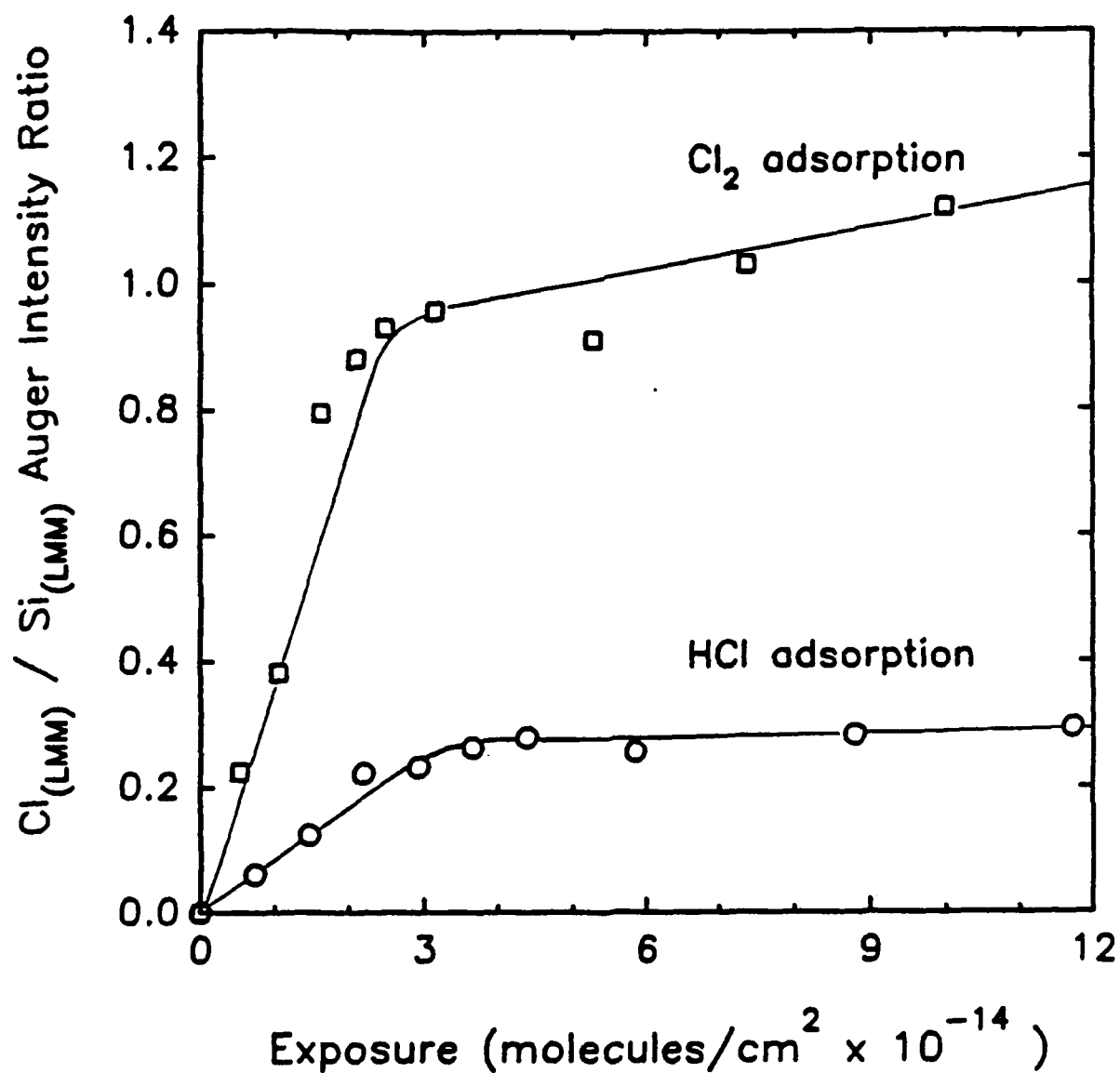
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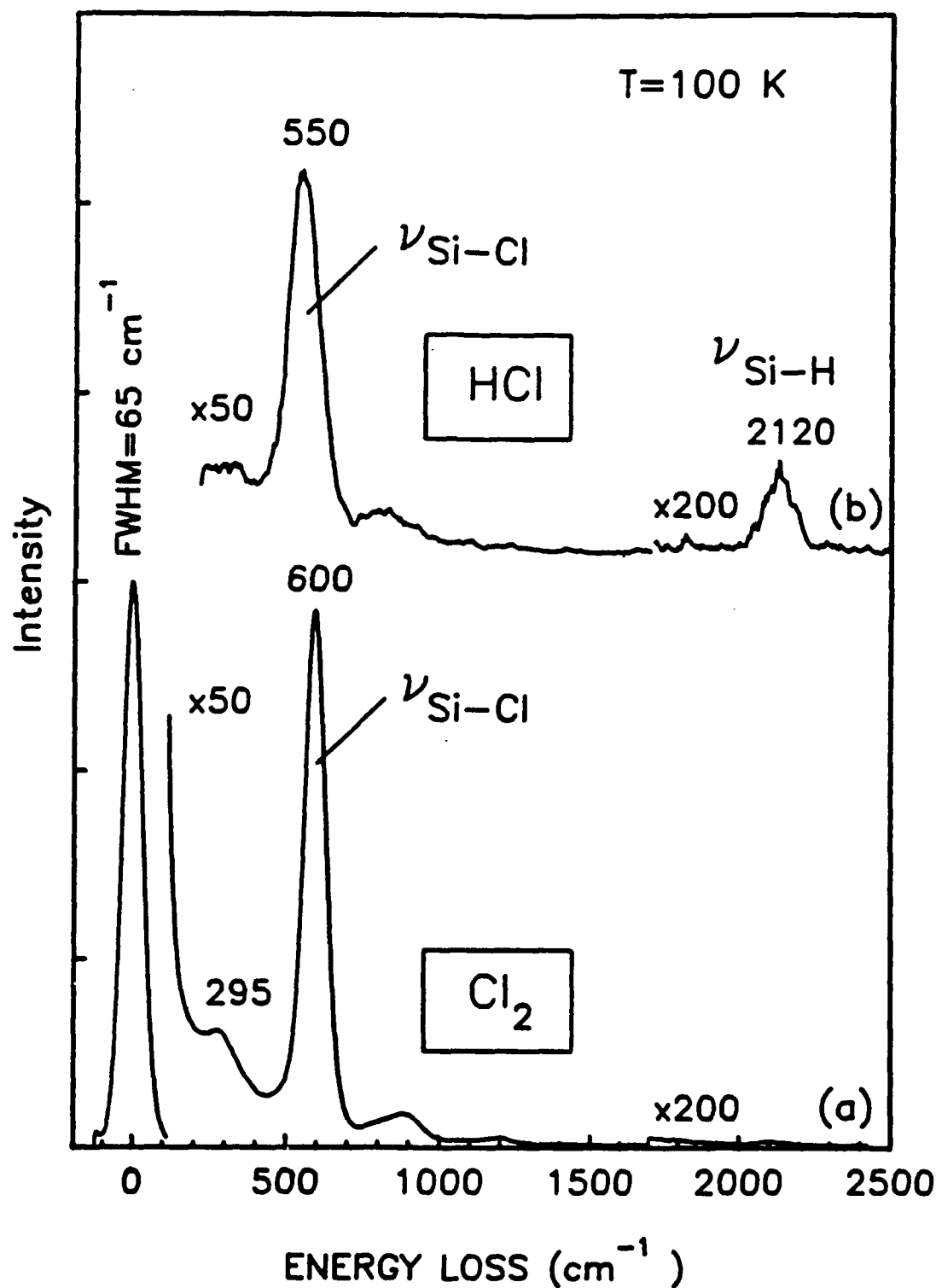
Figure Captions:

- Fig. 1. Rate of Cl coverage increase for Cl_2 and HCl adsorption on $\text{Si}(100)-(2 \times 1)$ at 100K using Auger Spectroscopy. $V_e = 2.0$ kV.
- Fig. 2. HREEL spectra of Cl_2 and HCl adsorption on $\text{Si}(100)-(2 \times 1)$ at 100K: (a) Cl_2 exposure= 7.4×10^{14} molecules/ cm^2 ; (b) HCl exposure= 8.7×10^{14} molecules/ cm^2 . Contour profiles are plotted by an increment of 1/6 of their peak maxima.
- Fig. 3. Cl^+ ESDIAD patterns of (a) Cl_2 (after annealing to 673K to get sharp monochloride pattern), and (b) HCl (120K adsorption). Near saturation coverages were achieved for Cl_2 and HCl adsorption prior to these measurements. $V_e=120$ eV.
- Fig. 4. Temperature programmed desorption from layers produced from Cl_2 and HCl adsorption to saturation coverage on $\text{Si}(100)-(2 \times 1)$. Heating rate is $\sim 3.6\text{K/sec}$.

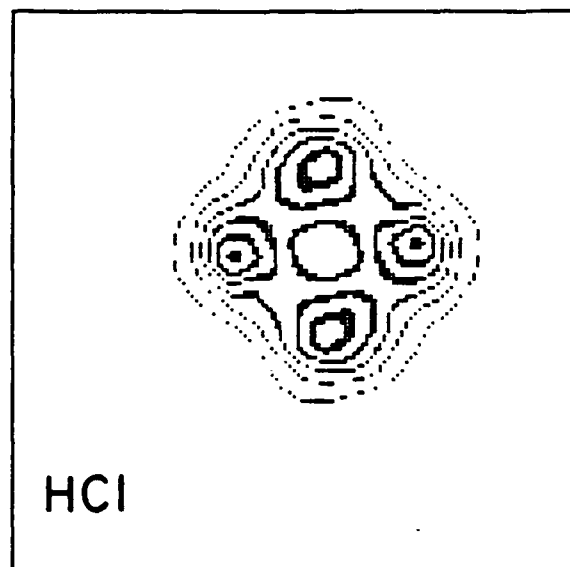
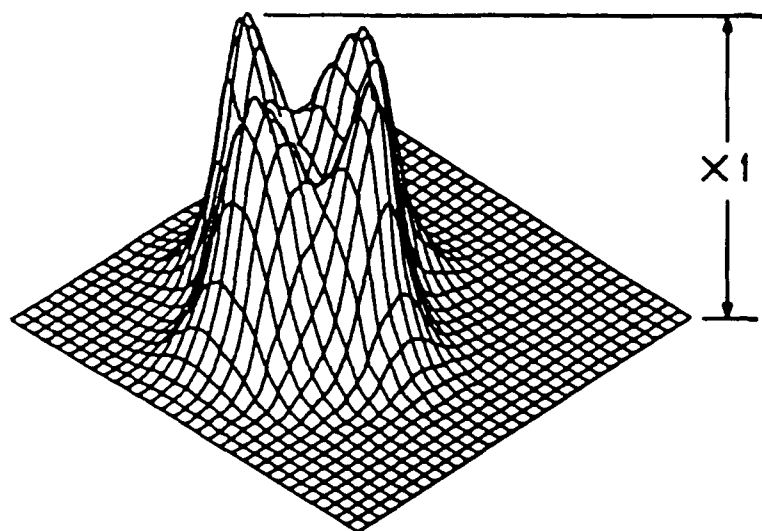
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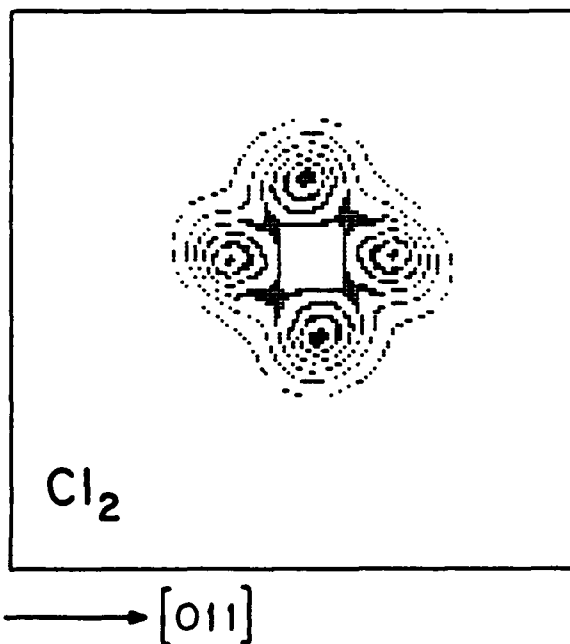
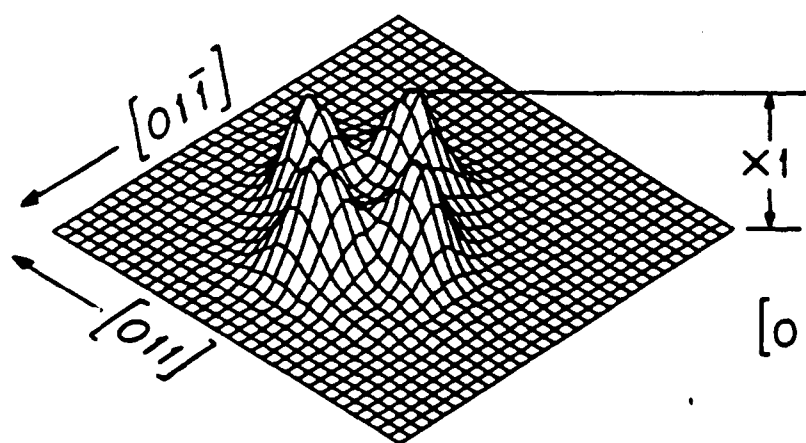
Comparison of the Vibrational Spectra
for Cl_2 and HCl Adsorption on $\text{Si}(100)$



Cl^+ ESDIAD Studies for Cl_2 and HCl Adsorbates on $\text{Si}(100)-(2 \times 1)$

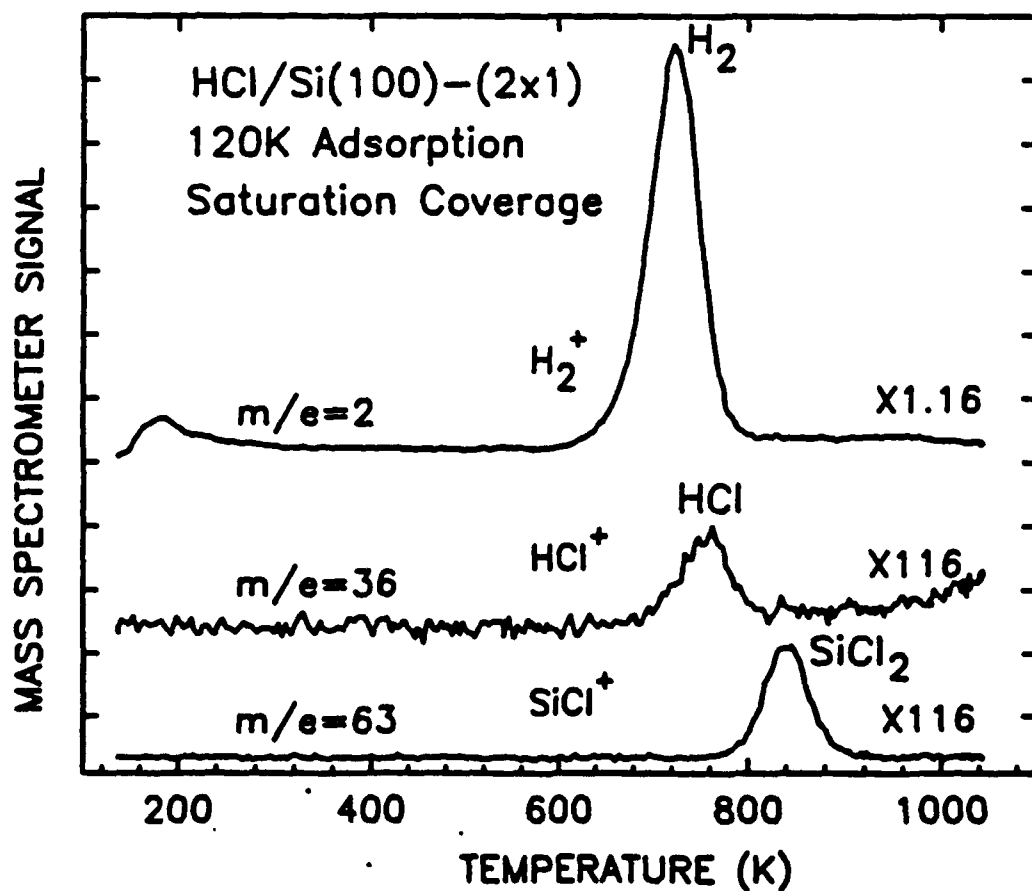
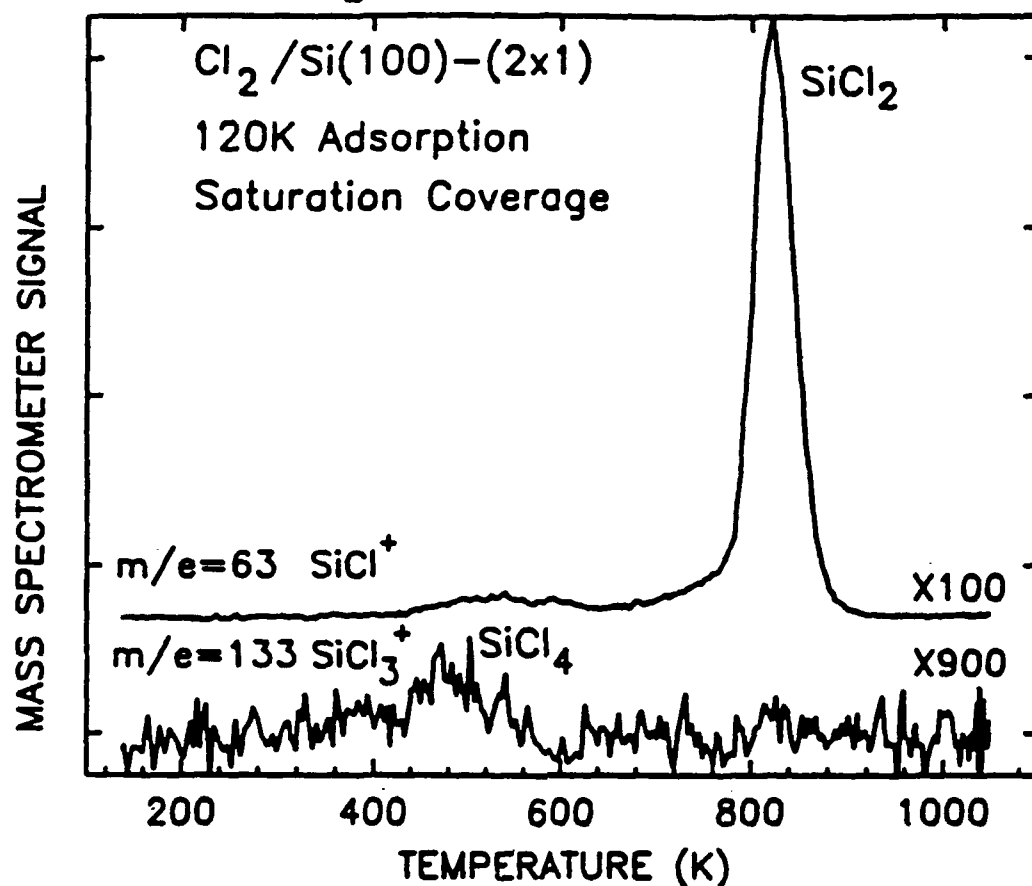


(b)



(a)

Comparison of Thermal Desorption
From Cl_2 and HCl on $\text{Si}(100)$



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