

行政院國家科學委員會專題研究計畫 成果報告

應用"染色法"研究晶體表面結構與複雜表面上反應的物理
機制(3/3)

計畫類別：個別型計畫

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執行單位：國立交通大學物理研究所

計畫主持人：林登松

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行政院國家科學委員會補助專題研究計畫 成果報告

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計畫主持人：林登松 教授

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吳依亭、黃乾廷、吳曉亭

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執行單位： 交通大學物理研究所

中 華 民 國 95 年 10 月 10 日

行政院國家科學委員會專題研究計畫成果報告

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一、進度與結果簡要說明

本年度我們主要研究重點在完成以穿隧效應顯微鏡STM之實空間影像來研究H在H/Si(100)-3x1 樣品上的熱脫附引起的結構相變化現象，此結果已發表。其他新領域的嘗試性工作如UHV non-contact AFM對SiO₂薄膜之研究也結束實驗工作，我們也繼續檢視STM之Cl₂, HCl, HBr/Si(100)-2x1 表面在吸附過程的實空間影像，探討各種反應模型，我們得到不少很有趣的資料，一部分也整理完成，準備送出發表。

以下是一部份完成的數據與分析：“Correlation of Reaction Sites during the Chlorine Extraction by Hydrogen-atom from Cl/Si(100)-2x1”

A. Introduction

The extraction of adsorbates on both metal and semiconductor surfaces by impinging hydrogen atoms has attracted much attention for dynamical understanding of the fundamental gas-surface reactions. Jackson⁰³, Quattr⁰⁵, Rettner⁹⁵, Buntin⁹⁸, Rettner⁹⁴, Koleske⁹³

One of the model systems among these studies is the production of HCl gas species from the Cl-terminated Si(100) surface (Cl/Si(100)). In this system, an incident H-atom flux would react with Cl atoms

adsorbed on the Si(100) surface and produce gaseous HCl molecules: $H_{(g)} + Cl_{(a)}/Si(100) \rightarrow HCl_{(g)} + Si(100)$. This gas-surface reaction has practical applications to Cl reduction in Si atomic layer epitaxy (ALE) at low temperature^{Gates_1992} and to dry etching processes in very-large-scale-integration (VLSI). One of the main scientific issues behind this is to examine the role of three disparate surface-reaction mechanisms at the gas/solid interfaces. In the idealized Langmuir-Hinshelwood (LH) mechanism, two reagents react after being chemisorbed and are in thermal equilibrium with the surface. Most surface reactions were believed to occur by this way. In the idealized Eley-Rideal (ER) mechanism, a direct, single gas-surface collision is responsible for a reaction between an incident gas-phase species and another adsorbed reagent. The occurrence of this pathway had been clearly demonstrated by Lykke and Kay^{Lykke90} and by Rettner. In the hot atom (HA) mechanism, a trapped incident gas-phase species bounces a few times or diffuses for a short distance before reacting with another adsorbed

reagent. This pathway falls between the two idealized pathways and has been shown to be the dominant reaction mechanism for the production of both H_2 and HCl in the reaction of H atoms on H- and Cl-covered metal surfaces.^{Quattr05,Jackson02}

Halogen and hydrogen atoms form strong bonds on the semiconductor surfaces and barely diffuse at near room temperature. Therefore, surface species likely retain their position after an extraction of halogen by an incident H atom occurs.^{Aldao2001} Utilizing Auger electron spectroscopy (AES) and temperature-programmed desorption (TPD) mass spectroscopy, Cheng et al found that the halogen removal rate by H(g) is first order in both the Cl/Br surface coverage (θ_{Cl}, θ_{Br}) and in the H flux (F_H).^{Cheng_1992} They also reported an activation energy of 0.91 meV per Cl removal and concluded that the H-extraction process follows an Eley-Rideal reaction mechanism where the surface reaction is mainly driven by the high internal energy of incident atomic hydrogen. Using time-of-flight scattering and recoiling spectroscopy (TOF-SARS) to measure the real-time surface H and Br coverage, Koleske and Gates verified that the removal rate of Br on the Si(100) surfaces with H atom has a linear dependence on both θ_{Br} and F_H below 500°C. In addition θ_{Br} and F_H , the same reaction on the Si(111) surface also depends linearly on the hydrogen coverage θ_H , suggesting that the reaction is second order.

The linear dependence of the reaction rate on θ_{Br} is consistent with an ER pathway.

However, the structure dependence of the reaction led to the suggestion that H atom may be partially accommodated at the surface in a mobile “hot precursor” state before the reaction with adsorbed Br. In the theoretical aspect, Kim, Ree, and Shin studied the H(g)+Cl(ad)/Si(100) system using the classical trajectory approach and concluded that all reactive events occur through the localized ER mechanism.^{kim1998}

As mentioned above, previous experimental studies had employed various spectroscopic techniques to measure the kinetics and dynamics of the gas-surface reaction. Hattori et al. had first studied that atomic hydrogen extracts chlorine from Si(111)-7 × 7 using scanning tunneling microscopy (STM).^{Hattori_98} The authors showed that Cl atoms are extracted from the Cl-covered Si(111) surface by atomic H, and the surface Si atoms after H bombardment are terminated with H atoms. The clean Si(100) surface after Cl termination at room temperature has a relative simple structure: the dimer bonds of silicon retain and the surface layer consists rows of Cl-Si-Si-Cl species.^{Lin_2002, Yate1995} Extraction of one or two Cl atoms on the Cl-Si-Si-Cl species and further H-bonding on the dangling bonds left behind, namely -Si-Si-Cl, -Si-Si-, H-Si-Si-, and H-Si-Si-H, also exhibit the same dimerized structure.^{Gao1993, Yate1995} Taking

these advantages, we utilized both the synchrotron radiation photoemission spectroscopy and STM to observe the Cl/Si(100) surface in atomic resolution after H-atom exposure. By comparing the results from the measurement with those from computer simulation, it is evident that the reaction does not occur through a single collision between the gas atom and the adatom.

B. Results and Discussion

3(a) Photoemission results

High-resolution synchrotron radiation core-level photoemission spectroscopy can be used to distinguish atoms at nonequivalent sites and in different chemical bonding configurations, according to shifts in their binding energy^{himpsel91}. Figures 1(a) and 1(b) show the respective surface-sensitive Cl $2p$ and Si $2p$ core-level spectra (circles), and their decomposition into constituent components from the Cl-Si(100)- 2×1 surface before and after H bombardment at 325 K for various dosage. All fitting was least-squares fitting^{chiang1998}. Each component that consists of a pair of spin-orbit split doublets is assumed to have the same Voigt line shape.

The Cl $2p$ spectra in Fig. 1(a) can be analyzed with a component that consists of a pair of split doublets separated by 1.60 eV. The binding energy of these Cl $2p$ spectra relative to that of the corresponding Si $2p$ remains at ~ 99.60 eV, suggesting that the Cl

atoms form similar Si-Cl bonds. Figure 2 plots the integrated intensities of the Cl $2p$ spectra (I_{Cl}), which is proportional to the surface Cl coverage. The integrated intensity of the bottom spectrum is normalized to be 1.0 because the chlorine coverage is nominally 1 ML for the Cl-saturated Si(100) surface before H-atom bombardment. I_{Cl} decreases linearly with the dosage of H-atoms at the early stage, indicating that Cl atoms were removed by impinging H atoms. This result is consistent with a previous study.

The bottom spectrum in Fig. 1(b) displays the Si $2p$ core level spectra for the Cl-Si(100)- 2×1 surface. This Si $2p$ spectrum consists of two components, B and Si⁺, separated by about 0.9 eV. The B component was responsible for emission from the bulk and the Si⁺ component from the surface Si-Cl species.^{Lin2003} As the exposure of atomic hydrogen increases, both the intensities of the Si⁺ component and the Cl $2p$ spectra drop off. This occurrence suggests that H atoms reduce the surface Cl coverage, as a previous report has found. After >1000 L of apparent exposure, the line shape of Si $2p$ is similar to that (top spectrum in Fig. 1(b)) obtained by direct, high-dosage hydrogen exposure on the clean Si(100)- 2×1 surface at room temperature.^{Yamamoto19} This observation indicates that hydrogen atoms terminate nearly all surface dangling bonds and form a mixture of dihydride and monohydride

surface when most Cl atoms are extracted. Note that a small component labeled Si^{2+} emerges in Fig. 1(b) after H impingement. The chemical shift of Si^{2+} , around 1.78 eV on the higher bonding energy side of B, is consistent with a charged state of +2 for Si atoms and responsible for SiCl_2 species. Presumably, the SiCl_2 species were formed as a consequence of the highly exothermic uptake of halogens during the extraction. Although more study is needed, the emergence of the dichloride species implies that impinging H atoms induce other surface reactions besides extracting upon collision with a surface adatom.

3(b) STM results

The clean Si(100) surface consists rows of dimers; the two dangling bonds from the two atoms in a dimer form a weak pi-bond.^{Boland1993} Cl adsorption on pure Si(100) surfaces saturates the dimer dangling bonds while preserving the basic (2x1) dimer structure without buckling as Fig. 3(a) displays.^{Lyubin1998, Xu2003} Notably an apparent dimer row in the empty state images is not formed by rows of Cl-Si-Si-Cl species, but the center part of two adjacent Cl-Si-Si-Cl species on two neighboring rows.

In Fig. 3(a), a handful of dark sites can be discerned to each occupy one side of a Cl-Si-Si-Cl species. As Figs. 3(b)-3(c) shows, the density of these dark sites increase with the H exposure. The dangling bonds

generated by the Cl removal exhibit higher apparent height due to enhanced tunneling near the Fermi level WeavorCl and are highly reactive for further H-adsorption. The dark sites in Fig. 3 are H-terminated sites. The initial H coverage on the Cl/Si(100) surface is less than 0.02 ML and are likely the residue during preparing the clean Si(100) surface and/or due to the adsorption of impurity HCl molecules in the Cl_2 gas source. The remaining coverage of surface monochloride sites can be obtained by direct counting in the STM images; the results are also plotted in Fig. 2. Since the STM and photoemission measurements were performed in different chambers with separate filaments, the actual H dosages for the two measurements are different but largely proportional as displayed in Fig. 2.

When the substrate temperature was held at RT during H-atom exposure, a reaction site, where a Cl atom was removed by a H atom and a H atom adsorbs subsequently, presumably undergoes no diffusion.^{Aldao2001, Owen1996} The brightest humps in the images are likely weakly bonded terrace SiCl_2 moieties, as evident from the photoemission spectra and discussed in the previous section. Besides the remaining Cl-terminated sites and bright humps, most of the reacted sites in Figs. 3(b)-(d) appear to be H-terminated. At the first glance, the H-terminated sites appear to be randomly dispersed. However, as will be analyzed and

discussed, the density and sizes of clusters grouped together by neighboring reaction sites appear to be larger than that created by random Cl extraction. At higher H-atom exposure, even two-dimensional islands with the H/Si(100)-2x1 structure, as Fig. 3(d) shows, can be easily found.

Figures 4(a) and 4(b) show the STM images for H(g)+Cl(ad)/Si(100) reaction at a substrate temperature of ~600 K. Similar isolated dark sites occupying one side of a dimer can be easily identified in Fig. 4(a); they are H-terminated sites after Cl-removal. Figure 4(b) shows that the density of the Cl-removal sites increases as the H-atom dosage and clustering of reactions sites become evident at higher H-atom dosage. These results are similar to that obtained at near room temperature.

4. Discussion

In the ER mechanism, a Cl-extraction reaction occurs via a collision induced desorption (CID). The calculated CID cross section is smaller than a unit cell within a small proximity around the point where a H atom strikes. The gas-phase H atoms impinge on the surface in a random fashion. If a Cl atom is removed upon a direct H-collision, the new reaction site is generated no matter what neighboring chemical environment surrounds the site. Specifically, the removal probability upon collision with a H atom is not changed when a Cl-Si surface species is neighboring to one or more dangling bond sites or monohydride sites. If this assumption is valid, the distribution of the Cl-removal sites by the random and

sequential impingement of gas-phase H atoms will be completely random in the STM images.

Figures 5(a), 5(b), and 5(c) display the results of reaction-site distribution from computer simulation based on this assumption. In the simulation, a reactive site was randomly generated based on the small impact parameters found in the classical trajectory approach. In the figures, the reaction sites are marked in different graded levels (0-8) for easy visual recognition of the degree of reaction-site clustering, as will be discuss later. The unnormalized pair distribution function (g') of reaction sites,

$$g'(j) = g(j)\theta = \frac{1}{N} \sum_{i=1}^N \frac{n_i(j)}{m(j)},$$

where $n_i(j)$ is the number of j th-nearest-neighbor reacted sites around the i th reacted site, θ the coverage, and $m(j)$ the number of j th-neighbor sites.^{trost1996}

As expected from the random sequential H-impingement, g' is roughly equal to the coverage of reacted sites, independent of the site index. The values of g' calculated from results of computer simulation such as Figs. (d-f) are in agreement with the expected values as Fig. 6 shows. In contrast, the pair distribution for sites $j=1-4$ obtained from STM images is boosted by about 20%, as displayed in Fig. 6. The pair distribution for sites $j=5-8$ is also boosted significantly at higher coverages. The meaning of the pair distribution function is that deviations from a random distribution of reaction sites. The deviations from $g'(j)=\theta$ corresponding to a random distribution suggests the existence of correlation and interaction between reaction sites, and therefore rules out the ER

process.

Another way to examine whether or not the clustering and islanding of reaction sites showing in the STM images results from random H-impingement, the STM images were digitized and displayed in Figs. 5(d), 5(e), and 5(f) in the similar fashion with Figs. 5(a-c) for direct comparison. A set of categories, denoted as 0-8, is assigned. The higher the category index, the higher degree of grouping clustering. Vision inspection of Fig. 5 suggests that the population for categories with higher category index (i) obtained from the STM measurement ($P_{\text{STM}}(i)$) is higher than that that ($P_{\text{Sim}}(i)$) from the simulated images. Their ratios $P_{\text{STM}}(i)/P_{\text{Sim}}(i)$, plotted in Fig. 7, deviate significantly from 1.0, especially for categories with category index higher than 4. This finding also indicates that the simulation based on the assumption of a pure ER process disagree with the experimental results. The cluster formation of reaction sites can only be realized if an impinging H atom would “probe” around the chemical environment in a small (HA) or large (LH) range beyond the collision spot. The sizes of clustering in Figs. 5(d-f) are not large; the initial pair correlation is limited to nearest 4 surrounding sites. These facts suggest that the reaction of Cl-extraction likely follows the HA process.

4. Conclusion

Distinguishing detailed surface reaction mechanism has been a difficult issue. The $\text{H}(\text{g})+\text{Cl}(\text{ad})/\text{Si}(100)$ is an important prototypical system for the study of the ER, HA, and LH mechanism. In our work, atomic resolved STM images, spectroscopic measurements of core level photoemission, and computer simulation together provide a detailed picture of the atomic processes involved in this seemingly simple gas-surface reaction. The core level measurement and STM images observed the formation of SiCl_2 surface species, indicating small fraction additional reactions occur in addition to the Cl removal upon impingement of H atoms. The Cl-removal sites obtained by analyzing the STM images were found to be correlated to near neighboring reacted sites. These experimental results cannot be explained by the pure Eley-Rideal process. We conclude that the HA process likely occurs during the atom-atom collision.

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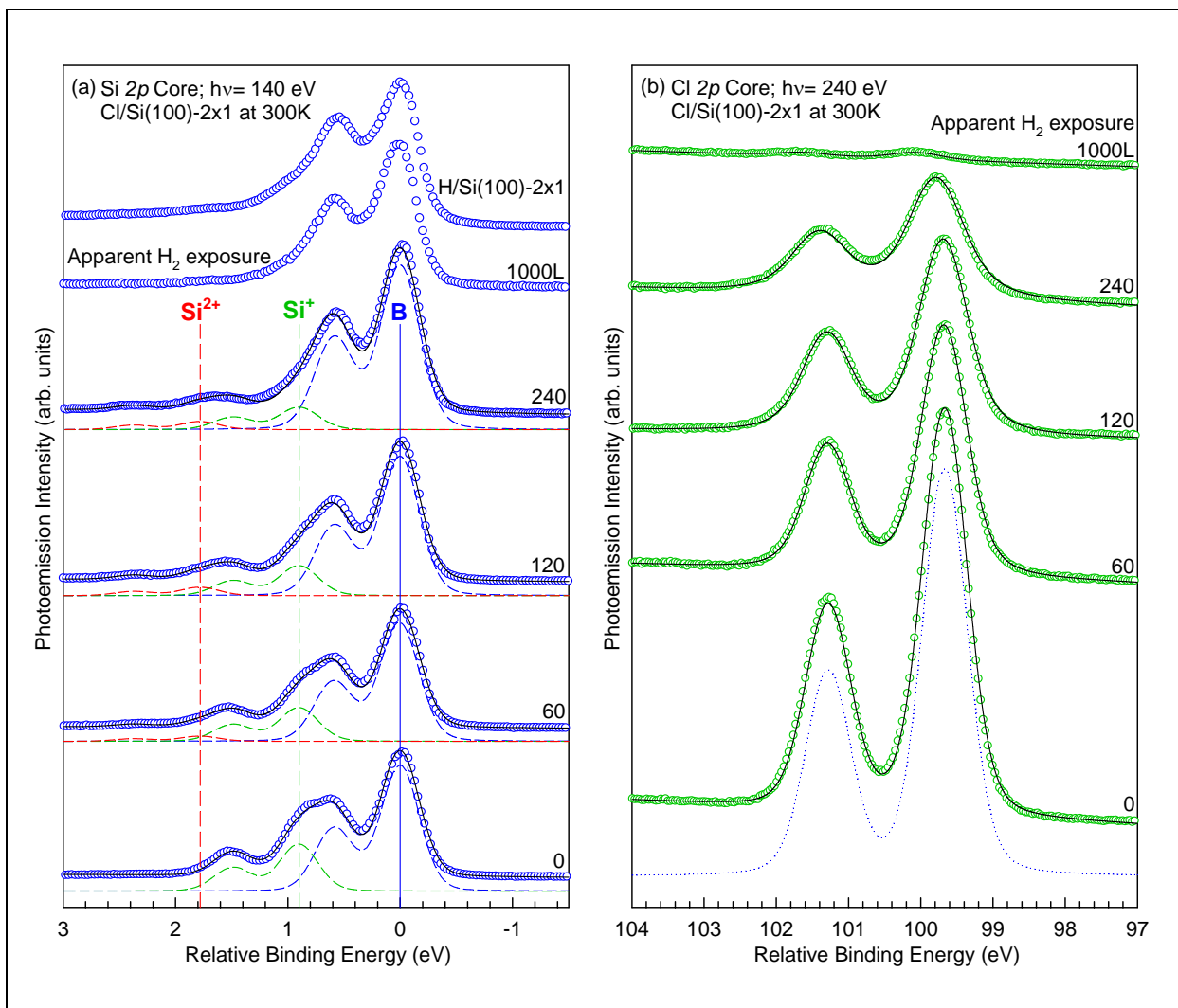


Fig. 1. The (a) Cl 2p and (b) Si 2p core level photoemission spectra (circles) for the Cl-Si(100)-2x1 surface and the same surface after various apparent H-atom dosages as labeled. The solid curves are fits to the spectra. The curves labeled B (black), Si⁺ (green dash) and Si²⁺ are the results of decomposition of the spectra into contributions from the bulk, Si-Cl, and Cl-Si-Cl species, respectively. The energy zero in (b) refers the 2p_{3/2} bulk position for the Cl-Si(100)-2x1 surface. To eliminate the band bending effect, the relative binding energy for the Cl 2p refers to the corresponding Si 2p_{3/2} line of the B component in (b).

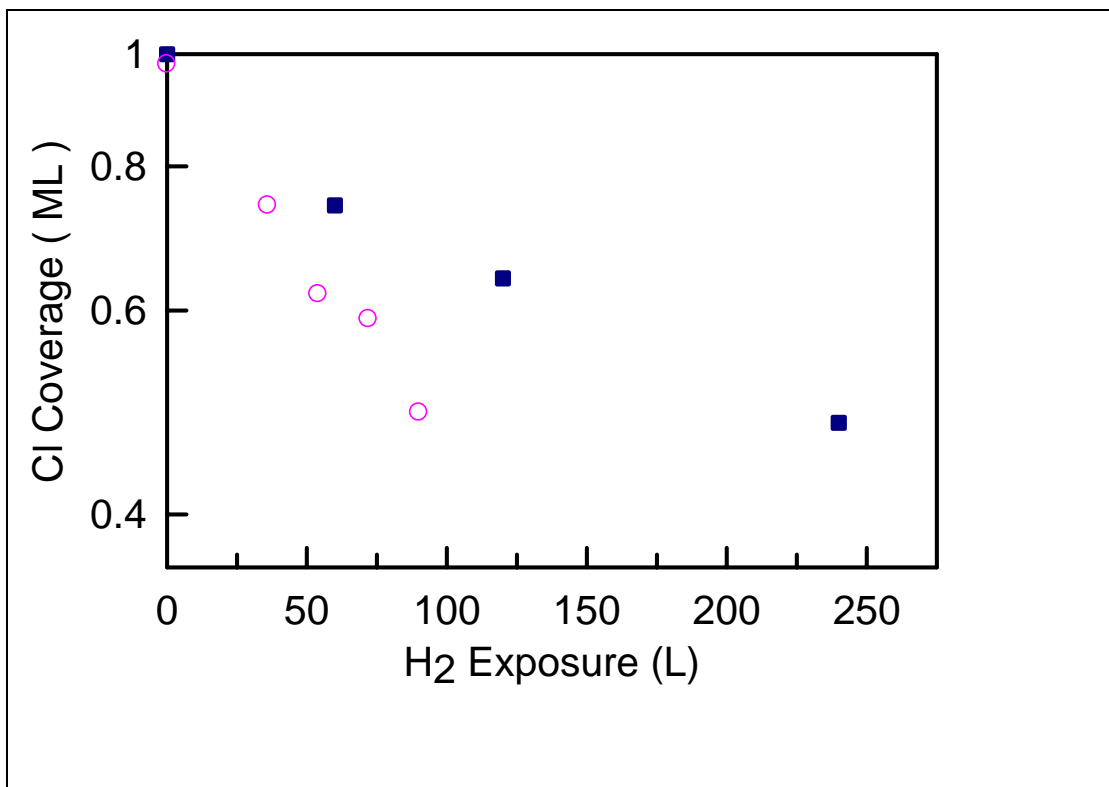


Fig. 2. Cl coverage calculated from the integrated intensities of the Cl 2p core-level spectra in Fig. 1(a) (solid squares) and from those counting from the STM images (open circles). The initial coverage is about 1.0 ML based on the STM result.

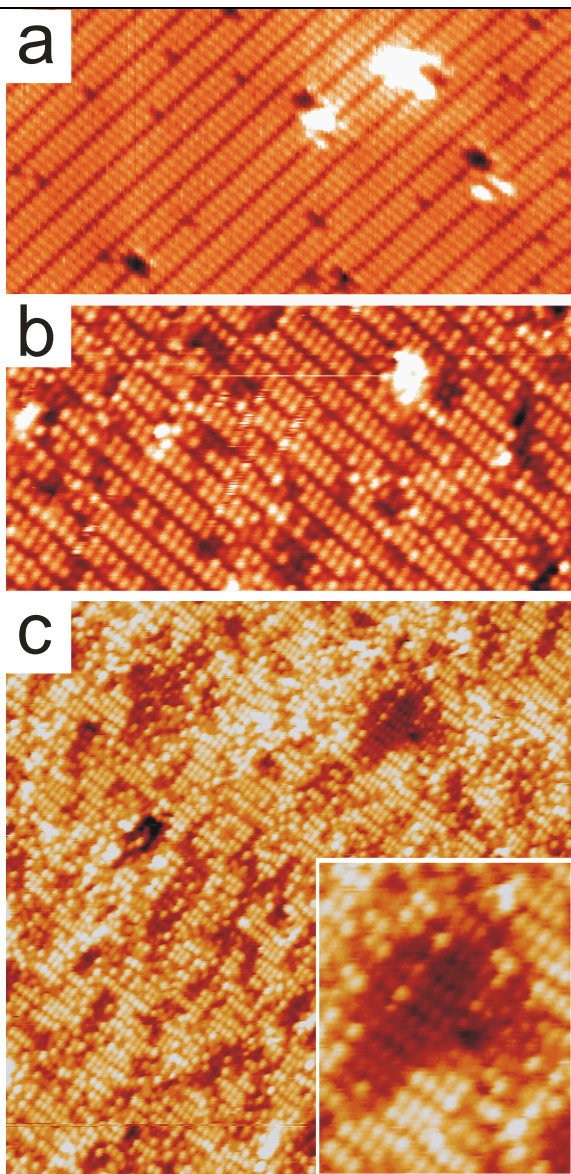


Fig. 3. STM images of the Cl/Si(100)-2x1 surface after (a) 0, (b) 20, (c) 90 L apparent dosages of H atoms. The sample bias used was +2 V. In (a) the green rectangle box, running from the upper left to the lower right, encloses a row of ten Cl-Si-Si-Cl (monochloride) species. A surface Cl atom appears as a bright protrusion and forms a narrow ellipse with another in the neighboring monochloride row in the image. The green and blue arrows point to a missing dimer defect site and a H-termination site, respectively. The inset in (c) shows a 2x1 area of nearly complete H-termination after Cl-removal.

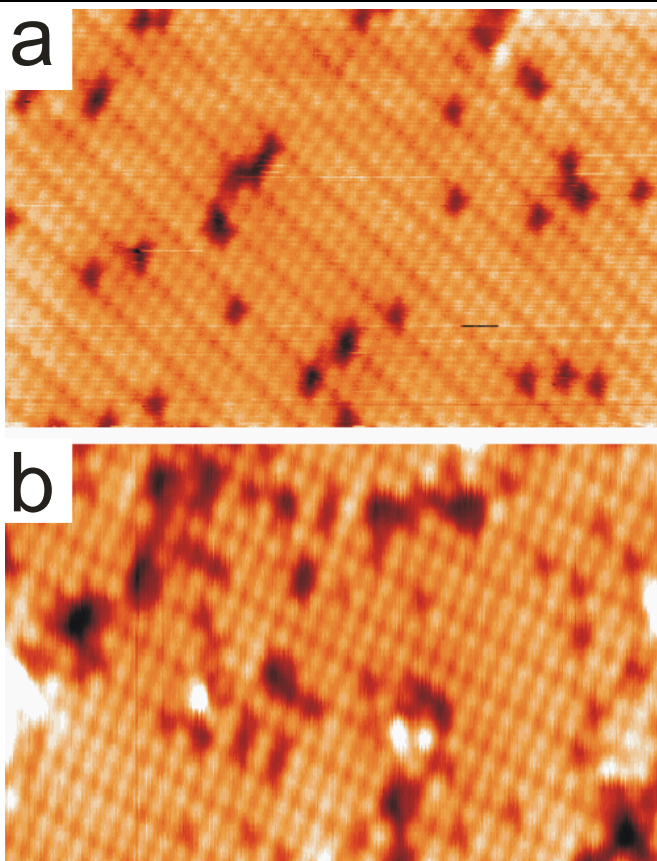


Fig. 4. STM images of the Cl/Si(100)-2x1 surface after (a)12 L, (b) 54 L apparent dosages of H atoms at a sample temperature of 600 K. The sample bias used was +2 V.

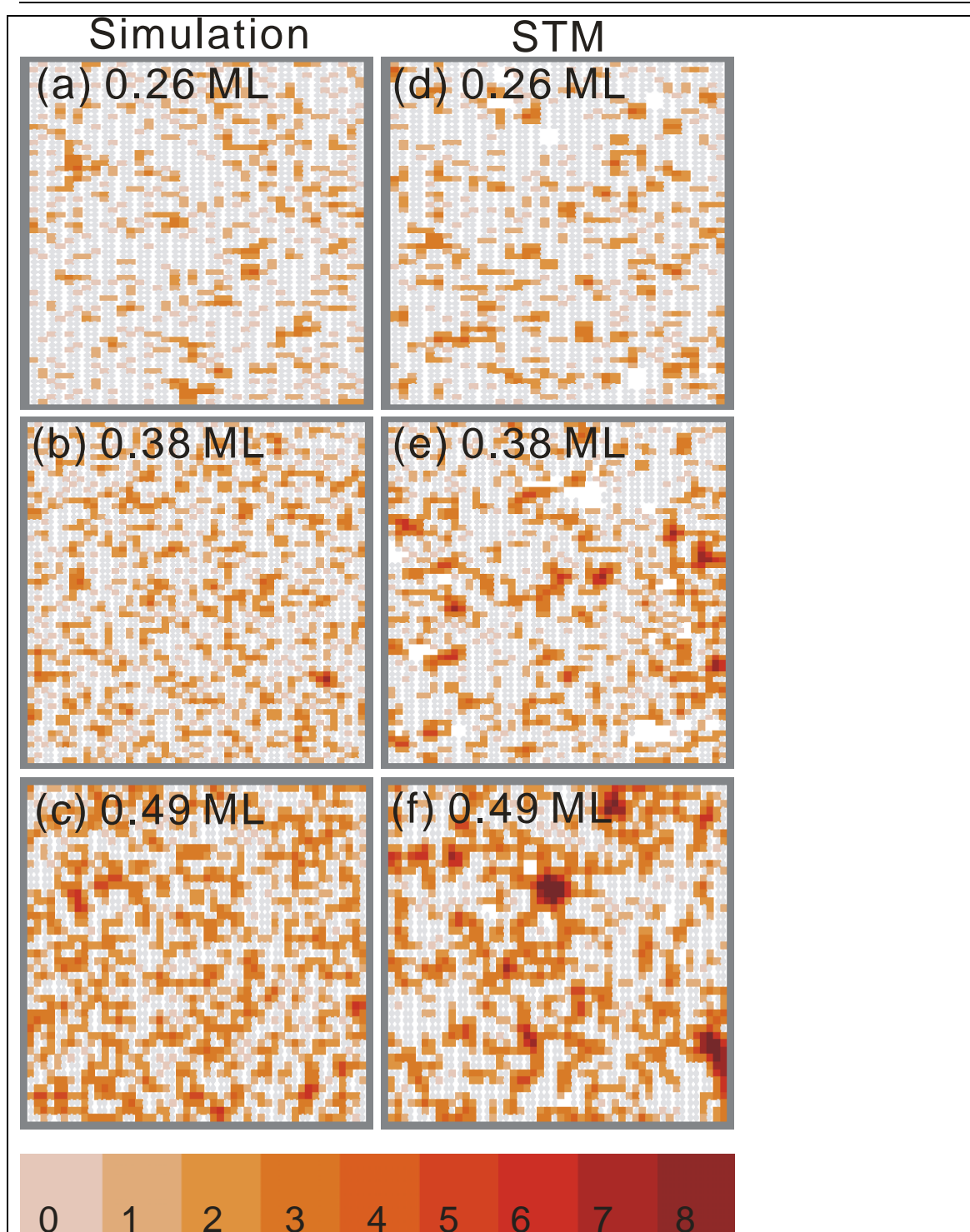


Fig. 5. Distribution of reacted sites obtained from (a-c) the results of simulation and (e-f) the STM images. The coverage of reacted sites in monolayer is labeled. Category 0, 1, 2, 3, and 4 are reaction sites with 0, 1, 2, 3, and 4 neighboring reaction sites among sites indexed 1-4, respectively. Category 5,6,7 and 8 are Category 4 reaction sites and have 1, 2, 3, and 4 neighboring Category 4 sites, respectively.

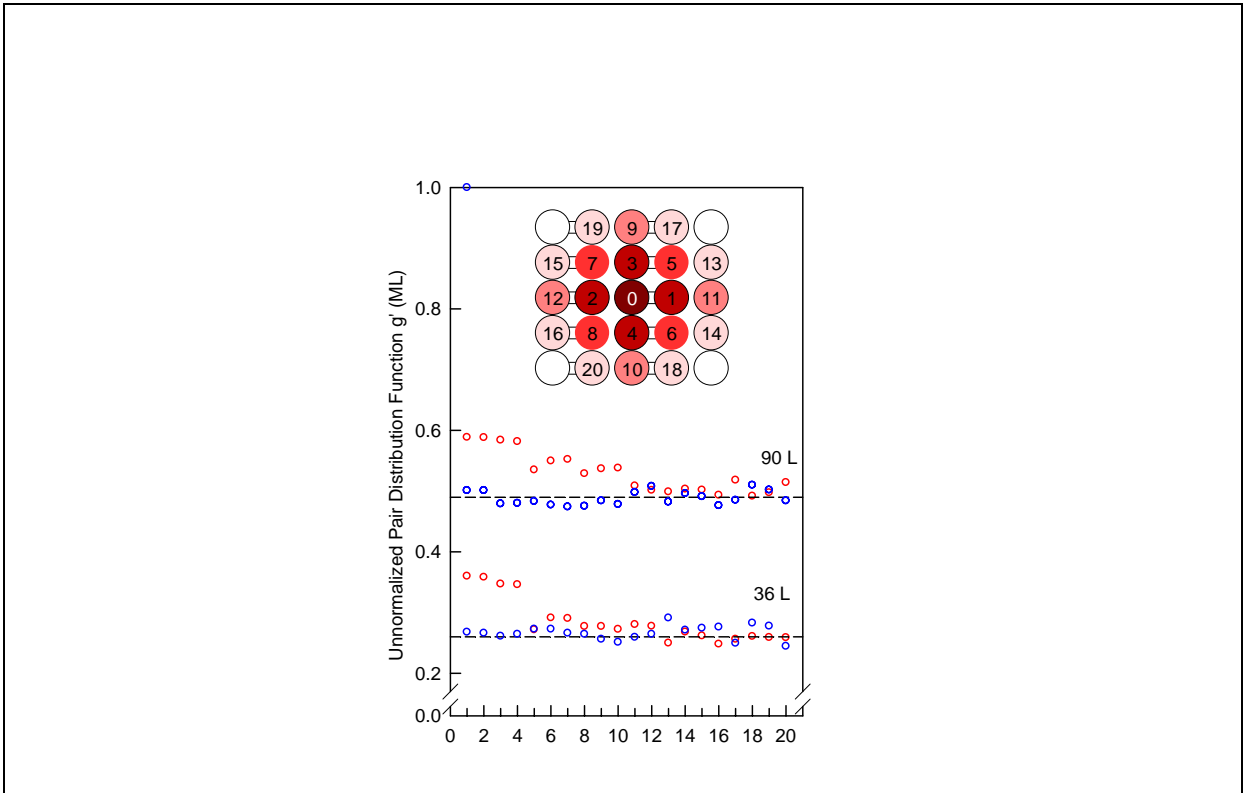


Fig. 6. The unnormalized pair distribution function of reaction sites vs the neighboring site j calculated from the STM images (red), the simulation (blue) and that expected from completely random distribution (dashed curves). The inset shows the Cl/Si(100)-2x1 lattice, each circle corresponding to a Cl adatom site. Numbers mark the index j of the distance between an Cl-removed site in the center and a Cl or Cl-removed site in the respective position.

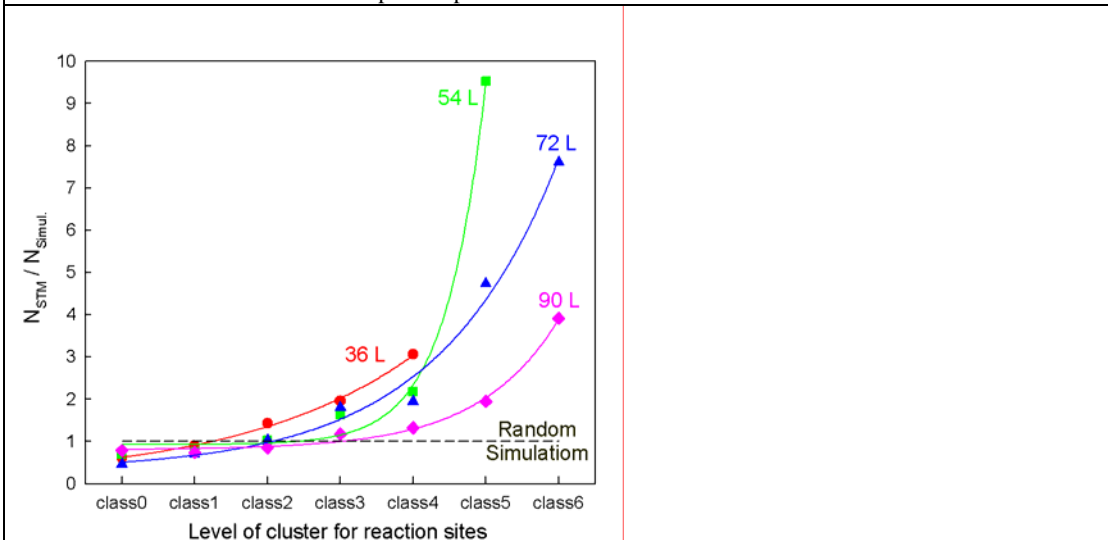


Fig. 7. The ratio of population between the measurement of STM $P_{STM}(i)$ and the result of simulation $P_{Sim}(i)$ vs category index.

出國報告

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會議時間: 自 95 年 7 月 30 日 至 95 年 8 月 4 日 2006

(一) 會議經過

1981 年掃瞄探針穿隧效應顯微技術之發明至今四分之一世紀 25 年, 諾貝爾獎頒發也已 21 年。這段期間蓬勃發展為數十種演申的瞄探針顯微、能譜技術, 現在已是重要科學發展之一支。而這類顯微、能譜技術為小尺度研究帶來圖突破, 也在十多年前引發所謂奈米科技的風潮。為了促進研究風氣與交流, 各國學界乃由輪流舉辦 NANO 國際會議, 本次是第九屆。國際掃瞄探針顯微術會議今年是第十四屆。由於研究學者重疊量大, 決定兩會議合併舉辦, 並重回掃瞄探針穿隧效應顯微技術之發明地瑞士舉辦, 以慶祝與紀念四分之一世紀前的發明。選在貝塞爾市市國際會議廳舉行乃是因為貝塞爾有非常強的掃瞄探針顯微技術研究與應用的研究陣容、人材濟濟。

本次繼承歷屆傳統, 包含內容廣汎, 論文質與量都可觀。會議共有來自近四十個國家, 60 個邀請演講, 近 1500 人參加, 一千餘篇論文發表。因為在瑞士舉辦, 與會者瑞、德最多。日本一樣是這行業研究的大本營, 雖地遠, 與會人也有近十分之一。

(二) 與會心得

由於與會口頭報告及壁報論文共近一千五百篇餘篇, 涵蓋 Scanning Probe Microscopy Instrumentation、Nanosystems, Nanomechanics and Nano-optics、Nanobiology, Nanomedicine、Molecular Electronics、Quantum Computing and Spintronics、Materials 四大領域、四十個子題, 範圍廣且多專業內容。筆者不認為有在此詳述之必要, 僅報告下列較印象深刻之觀察:

1. 以一般掃瞄探針顯微技術而言, 在會場上幾乎所有歐洲相關公司皆帶實物參展。器材與裝置皆已相當成熟, 硬體變化已不大, 但控制系統已走向全數位訊號, 即使連回饋訊號都不再使用類比電路, 顯見傳統電子學的應用將會有越來越大的改變。
2. 目前在 SPM 技術方面學術界仍不斷精益求精, 近來學界主要的興趣是原子解析度的非接觸探針掃瞄技術穩定性上面的發展, 德國已有團對嘗試撇開雷射光學槓桿偵測, 而直接以壓電管上作回饋訊號來源, 未來發展可能更新整體產業的技術。另一方面, 接觸探針掃瞄技術看到原子各種的解釋仍受到不少質疑與挑戰。
3. 以表面、薄膜、奈米科學研究而言, 今年的發表論文顯示, 整體研究穩定發展, 特別的新方向主要是超低溫、高磁場下的物理。尤其是所謂自旋解析掃瞄探針顯微技術已

有很穩定的發展，與超導物理研究，世界上已有很多的研究組進行此領域。這領域進入障礙高，須要高品質人力與物力，可是帶來新發現的機會也高，國內的研究環境與能力是難以支撐這種研究。

4. 一向熱門的所謂奈米生命科學方向，也有不少論文。整體而言，可以說仍是一個嘗試期。距成為生命科學主流或前導的目標尚有一段距離。

(三) 攜回資料

1. 會議論文摘要彙編。