

STM characterization of the Si-P heterodimer

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We use scanning tunneling microscopy (STM) and Auger electron spectroscopy to study the behavior of adsorbed phosphine (PH₃) on Si(001), as a function of annealing temperature, paying particular attention to the formation of the Si-P heterodimer. Dosing the Si(001) surface with ~ 0.002 langmuirs of PH₃ results in the adsorption of PH_x ($x=2,3$) onto the surface and etching of Si to form individual Si ad-dimers. Annealing to 350 °C results in the incorporation of P into the surface layer to form Si-P heterodimers and the formation of short one-dimensional Si dimer chains and monohydrides. In filled state STM images, isolated Si-P heterodimers appear as zigzag features on the surface due to the static dimer buckling induced by the heterodimer. In the presence of a moderate coverage of monohydrides this static buckling is lifted, rendering the Si-P heterodimers invisible in filled state images. However, we find that we can image the heterodimer at all H coverages using empty state imaging. The ability to identify single P atoms incorporated into Si(001) will be invaluable in the development of nanoscale electronic devices based on controlled atomic-scale doping of Si.

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I. INTRODUCTION

Currently there are several proposals to use phosphine gas (PH₃) to create atomic-scale devices by positioning P atoms on a surface using H lithography.¹⁻³ One such idea² involves the fabrication of a two-dimensional array of P atoms in Si for the realization of a silicon based quantum computer.⁴ A requirement of each of these schemes is that the P atoms, relinquished from the PH₃ molecules, are incorporated into substitutional lattice sites. Since PH₃ is used for doping Si in the semiconductor industry, the interaction of PH₃ with the Si(001) surface has been studied for over two decades using a wide variety of surface-sensitive techniques (e.g., Ref. 5). It is well established that the adsorption of PH₃ on the Si(001) surface is partially dissociative, resulting in the adsorption of both PH₃ and PH₂.^{3,6-10} Moreover, it is known that annealing PH₃ dosed surfaces causes the complete dissociation of the adsorbed PH₃ and PH₂ molecules (estimates of the temperature at which this occurs vary within the range 300–425 °C (Refs. 6,8,9, and 11) resulting in the incorporation of P into the surface to form Si-P heterodimers.^{3,8,12,13} The majority of the investigations mentioned above have studied the interaction of phosphine with the Si(001) surface at high surface coverages. However, at low coverages there are only two papers reporting detailed atomic-resolution scanning tunneling microscopy (STM) investigations of the Si(001):PH₃ system, the first by Wang *et al.*¹⁴ and the second by Kipp *et al.*¹⁵ While the experimental results presented in these two papers are in good agreement, their interpretations are contradictory.

The experimental results of Wang *et al.*¹⁴ and Kipp *et al.*¹⁵ showed that after the Si(001) surface was exposed to a low dose of PH₃, two types of bright features appeared on the surface. Both features were centered on top of the Si dimer rows, with one feature larger than the other. Wang *et al.*¹⁴ also observed an increase in the number of dimer vacancies on the surface after PH₃ dosing. In both studies, after the dosed surfaces were annealed (to 230 °C, Ref. 14, and to 350 °C, Ref. 15), the two types of bright features

disappeared from the surface and were replaced by one-dimensional (1D) chains running perpendicular to the underlying Si dimer rows and a few small two-dimensional (2D) islands. The above data were interpreted by Wang *et al.*¹⁴ in the following way: the larger bright feature was a single Si atom (a monomer) ejected from the surface and the smaller bright feature was a PH₃ molecule adsorbed on top of a Si dimer. Upon heating the surface to 230 °C the Si monomers combined to form 1D rows and small 2D islands. This annealing also induced complete PH₃ dissociation, with the P atoms being incorporated into the surface and the H adsorbed onto the surface to form monohydride dimers. Kipp *et al.*¹⁵ presented a different interpretation of the above data. They concluded that the large bright features were PH_n ($n=1-3$) molecules adsorbed at defects and the small bright features were P-P dimers, resulting from the pairing and subsequent dissociation of two PH₃ molecules (with the dissociated hydrogen atoms also bonding to the surface). Upon heating the surface to 350 °C Kipp *et al.*¹⁵ concluded that the P-P dimers coalesce to form small chains and islands. They suggested that the formation of large islands was hindered by the presence of adsorbed H on the surface.

In contrast to the results of Wang *et al.* and Kipp *et al.*, separate Fourier-transform infrared spectroscopy studies⁹ and a combined electron energy-loss spectroscopy and thermal programmed desorption study⁶ have indicated that molecular PH₃ adsorption is accompanied by a partial dissociation of PH₃ to form adsorbed PH₂ and H, during phosphine adsorption at room temperature. Therefore, further study of this system is required to consolidate the different interpretations. We have used high-resolution STM and Auger electron spectroscopy to study the evolution of a PH₃ dosed Si(001) surface, as a function of temperature and coverage. Particular emphasis was placed on the process of P incorporation into the Si(001) surface and the formation of the Si-P heterodimer. To this end we have also performed a study of surface segregated P from a Si encapsulated δ -doped layer. Pivotal to these experiments is the ability to resolve the presence of a single P atom in the Si(001) surface using STM.

We show that the isolated Si-P heterodimer appears as a zig-zag feature in filled state STM images, but that the heterodimer is not visible if there is a moderate coverage of monohydrides on the surface. However, empty state imaging can be used to identify the heterodimers at all coverages.

II. EXPERIMENT

Experiments were performed in ultrahigh vacuum (base pressure $<5 \times 10^{-11}$ mbar) using an Omicron variable temperature STM and electrochemically etched tungsten tips. The *n*-type Si(001) sample had a resistivity of $\sim 1 \Omega \text{ cm}$ and was prepared in vacuum by outgassing overnight at 575°C using a resistive heater element, followed by flashing to 1175°C by passing a direct current through the sample. After flashing, the samples were cooled slowly ($\sim 3^\circ \text{C/s}$) from 900°C to room temperature. For all anneal steps the sample temperature was maintained at the stated anneal temperature for 10 sec unless stated otherwise. The PH_3 dosing was performed by opening a precision leak valve between the UHV chamber and a PH_3 microdosing system. All doses were performed by back-filling the chamber with a 1×10^{-9} Torr pressure of PH_3 and controlling the dose in langmuirs [$1 \text{ L} = 10^6 \times \text{pressure (torr)} \times \text{dose (seconds)}$] via the dose time. Since the leak valve faced the sample, the absolute PH_3 dose may differ somewhat from other published doses, and our quoted doses should be considered as relative values. The ionization gauge was not in line of sight of the sample. All STM images were taken at room temperature and are filled state images (negative sample bias) unless stated otherwise. Dual bias STM images, where filled and empty state images are obtained simultaneously, were taken with a negative (positive) sample bias while scanning the tip in forward (reverse) direction. Auger electron spectroscopy (AES) data were obtained using an incident electron beam of 2 kV, sample current of $40 \mu\text{A}$, and a spot size of 1 mm. The Auger electrons were collected using a four-grid retarding field analyzer.

III. RESULTS AND DISCUSSION

A. Identification of surface features for room-temperature adsorption of PH_3 on Si(001)

Figure 1 shows STM images of the Si(001) surface after exposure to low coverages of phosphine (PH_3) gas, at room temperature. The clean Si(001) surface consists of rows of Si dimers which are seen as the parallel lines running diagonally across the images (along the $\langle 1\bar{1}0 \rangle$ direction). Figures 1(a) and 1(b) show a $21 \times 17 \text{ nm}^2$ area of a Si(001) surface before and after dosing with 0.002 L of PH_3 , respectively. The circled features in (a) are single dimer vacancy defects which are inherent to the clean surface and which we identify to distinguish them from species resulting from PH_3 dosing. After PH_3 dosing [Fig. 1(b)] we find that additional features have appeared on the surface, which are (i) adsorbed PH_3 , (ii) adsorbed $\text{PH}_2 + \text{H}$, (iii) dimer vacancies (in addition to those present before dosing), and (iv) Si ad-dimers. Figure 1(c) shows an image of a PH_3 molecule which has a height/diameter of $0.9 \text{ \AA}/11 \text{ \AA}$ in filled state images and is adsorbed

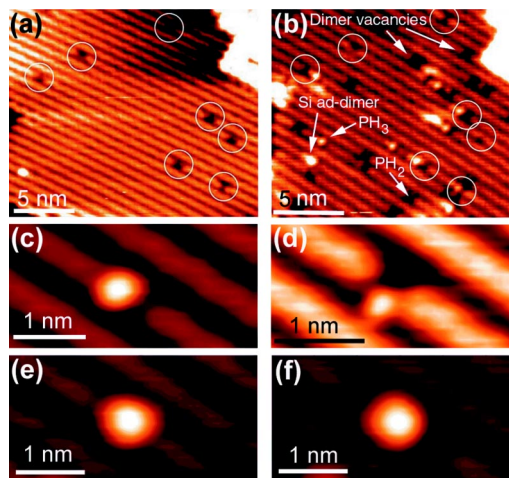


FIG. 1. STM images of the same area of a Si(001) surface (a) before and (b) after dosing with 0.002 L of PH_3 at room temperature. Imaging conditions were -1.6 V sample bias and 0.2 nA tunneling current. (c) and (d) show PH_3 and $\text{PH}_2 + \text{H}$, adsorbed on Si(001), respectively. Imaging conditions were -1.8 V sample bias and 0.1 nA tunneling current. High-resolution STM images of a Si ad-dimer are shown for (e) filled state (-1.6 V , 0.1 nA) and (f) empty state ($+1.2 \text{ V}$ and 0.1 nA) imaging.

upon the center of a dimer row, as previously identified by Wang, Shan, and co-workers.^{9,14} It can be seen in Fig. 1(c) that this molecule is adsorbed adjacent to a defect, which we find is commonly the case at low coverage. We believe that defects stabilize the adsorption of the PH_3 molecules centered over Si dimers. However we cannot rule out the possibility that the feature is a PH_2 radical centered on the dimer row, as suggested by Lin *et al.*¹⁰ Figure 1(d) shows a PH_3 molecule after it has dissociated into $\text{PH}_2 + \text{H}$ on a single Si dimer, as described.³ The observation of both adsorbed PH_3 in its molecular form, and as the dissociation products $\text{PH}_2 + \text{H}$, is in agreement with Fourier-transform infrared⁹ and photoemission¹⁰ results.

The third type of features in Fig. 1(b) are single and multiple dimer vacancies, resulting directly from the removal of Si from the surface layer during PH_3 dosing. While dimer vacancies are difficult to distinguish from monohydride dimers (formed when each Si atom of a dimer is terminated with a H atom) the formation of monohydride dimers is not expected at room temperature since single H atoms have insufficient mobility to diffuse and pair up into monohydrides.¹⁶ We assign the fourth feature to be a Si ad-dimer, centered on the dimer rows with a height/diameter of $1.3 \text{ \AA}/13 \text{ \AA}$ in the filled state image and a height/diameter of $1.9 \text{ \AA}/13 \text{ \AA}$ in the empty state, in agreement with the observation of Si ad-dimers by Swartzentruber¹⁷ and shown in Figs. 1(e) and 1(f), respectively. The filled state height of the feature (1.3 \AA) is of the same order as the distance between $[001]$ planes of the Si crystal (1.36 \AA) as expected for a Si ad-dimer. We discount the suggestion of Wang *et al.*¹⁴ that the feature is a Si monomer, since Si monomers are not observed in filled state images.¹⁸ We also rule out the fourth feature being either adsorbed P or PH species because Fourier-transform infrared spectroscopy⁹ and photo-

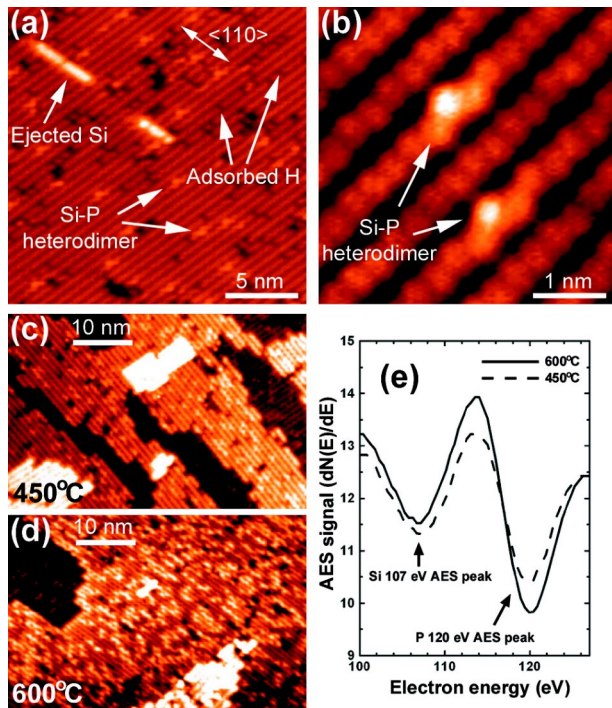


FIG. 2. (a,b) STM images of a Si(001) surface after dosing with 0.002 L of PH_3 and annealing to 350 °C at low and high resolution, respectively. (c,d) STM images of a Si(001) surface after saturation dosing with PH_3 , annealing to 600 °C, overgrowing with 5 ML (monolayer) of Si at room temperature and then annealing to (c) 450 °C and (d) 600 °C. (e) Auger electron spectra obtained from the surfaces shown in (c) and (d).

emission¹⁰ results show that these species are not formed after room-temperature PH_3 adsorption on Si(001).

B. P identification using STM and AES

We now describe the incorporation of P atoms into the surface, to form Si-P heterodimers, upon annealing. Figure 2(a) shows a Si(001) surface that has been exposed to 0.002 L of PH_3 and annealed to 350 °C. Three distinct types of feature are present on the surface, Si-P heterodimers, ejected Si dimer chains, and monohydride dimers. The ejected Si chains appear as short bright lines running along the $\langle 110 \rangle$ direction, perpendicular to the dimer rows. These features have the same height above the surface (1.36 Å) as a monolayer Si step¹⁹ and have a characteristic minima running lengthways along their center in empty state images [see Fig. 4(d)], indicating that they are indeed Si dimer chains as suggested by Wang *et al.*¹⁴ and not P dimer chains as proposed by Kipp *et al.*¹⁵ The monohydride dimers (H-Si-Si-H) appear as dark features, similar in appearance to missing dimer defects. Figure 2(b) shows a high-resolution image of two Si-P heterodimers, which appear as zigzags along the dimer row, with the highest part ~ 0.3 Å above the surface dimers.¹³ This zig-zag appearance indicates there is static buckling of the surface dimers on either side of the Si-P heterodimer. Figure 2(b) shows that this buckling decays in amplitude as a function of distance from the Si-P heterodimer and extends 2-3 dimers away from it.

To confirm the interpretation of Fig. 2(a) presented above, we compare the number of Si-P heterodimers in STM images with the fraction of P at the surface measured by Auger electron spectroscopy (AES). In order to control the number of Si-P heterodimers at the surface without the complication of ejected Si dimer chains and adsorbed H [Fig. 2(a)], we have created a buried P δ -doped layer and then diffused varying amounts of the P atoms to the surface using a range of anneal temperatures. The δ layer was produced by saturation dosing a Si(001) surface with PH_3 , annealing to 600 °C, and encapsulating with 5 ML (monolayer) of Si at room temperature.²⁰ Upon the annealing of this sample the buried P atoms diffuse to the surface at a rate determined by the anneal temperature. Figures 2(c) and 2(d) show STM images obtained after annealing the P δ -doped sample for 60 s at 450 °C and 600 °C, respectively. Figure 2(e) shows the 120 eV P AES peak obtained from the two surfaces. There is a direct correlation between the number of zigzag features in the two STM images and the intensity of the 120 eV P Auger peak, confirming that the zigzag feature contains P atoms. From the zigzag appearance we conclude that each of these features contain only one P atom since P-P dimers are known to have mirror symmetry about a line running along their center¹² and a feature consisting of neighboring incorporated P atoms *along* a dimer row would show neighboring atoms with similar intensities.

While the results presented here characterize the isolated Si-P heterodimer, the observation that P atoms incorporate into the Si(001) surface upon annealing fits well with the interpretation of Wang *et al.*¹⁴ of the annealing of the low dosed $\text{PH}_3/\text{Si}(001)$, and contradicts the interpretation of Kipp *et al.*¹⁵ We therefore conclude, following Wang *et al.*, that the processes that occur as a result of the 350 °C anneal are as follows; the PH_x ($x=2,3$) molecules first undergo complete dissociation. Each P atom then undergoes a substitution reaction with one atom of the Si surface dimer to form a Si-P heterodimer in the plane of the surface, thereby ejecting the displaced Si atom onto the surface. At 350 °C the displaced Si atoms are mobile enough to diffuse short distances²¹ and subsequently form 1D dimer chains on the surface. The H atoms liberated from PH_x dissociation are highly mobile at 350 °C and pair up to form the energetically favorable monohydride dimers.⁹

C. The changing appearance of the Si-P heterodimer with coverage

We have found that the appearance of the Si-P heterodimer varies greatly as a function of initial PH_3 coverage. Figure 3 shows a Si(001) surface after exposure to two different doses (0.002 L and 0.01 L) of PH_3 and subsequent annealing of the dosed surfaces to 350 °C and then 500 °C. If we compare the two 350 °C anneals, Figs. 3(a) and 3(b), we see that the surface that was exposed to 0.01 L of PH_3 (the higher dose) has significantly more Si chains and the onset of 2D Si island formation [Fig. 3(b)], as expected. After both the 0.002 L and 0.01 L dosed surfaces have been subject to a further anneal of 500 °C the Si chains have now disappeared, see Figs. 3(c) and 3(d). This can be explained since at 500 °C the diffusion of Si atoms on the surface is

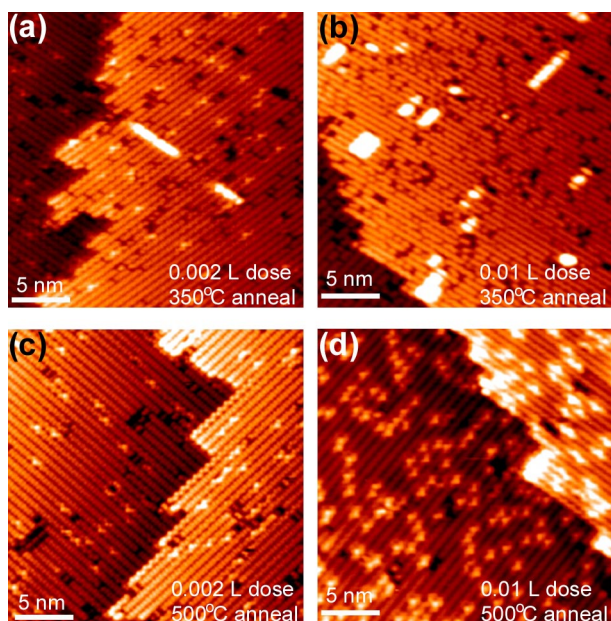


FIG. 3. (a), (b) STM images of a Si(001) surface heated to 350 °C for 10 s, after a PH_3 dose of (a) 0.002 L and (b) 0.01 L. (c) and (d) show the same surfaces in (a) and (b), respectively, after a further 10 s anneal to 500 °C. All images were obtained using a tunneling current of 0.13 nA and sample biases of -1.6 V except (d) which was -0.8 V.

sufficient such that all the Si ad-dimers migrate to step edges to which they preferentially bond. We now consider what information we can determine about the Si-P heterodimers, from Fig. 3. If we just consider Figs. 3(c) and 3(d), which show the 0.002 L and 0.01 L dosed surfaces after annealing to 500 °C, the data fit our explanation of the evolution of the PH_3 dosed Si(001) surface, i.e., the more PH_x ($x=2,3$) initially adsorbed on the surface, the more Si-P heterodimers are observed on the surface after the 500 °C anneal. However, the 350 °C anneals of the 0.002 L and 0.01 L dosed surfaces are not so easy to understand. We repeatedly observe that the 0.002 L dosed, 350 °C annealed surfaces such as that of Fig. 3(a) show moderate densities of Si-P heterodimers and monohydrides, as expected. Conversely, the 0.01 L dosed, 350 °C annealed surfaces such as that of Fig. 3(b) appear to show a high density of monohydrides and no Si-P heterodimers. We know from the 500 °C anneal of the 0.01 L dosed surfaces that Si-P heterodimers are abundant, so we can conclude that a high density of monohydrides inhibits the observation of Si-P heterodimers with the STM. It is known that monohydride dimers exhibit very little buckling²² and thus pin neighboring Si dimers in an unbuckled geometry. Hence, if the density of monohydrides is sufficiently high, it is reasonable to assume that almost all the surface Si dimers are pinned, even those in the vicinity of Si-P heterodimers. As a consequence, the characteristic zig-zag appearance of the Si-P heterodimer will be suppressed. It is now interesting to look again at Fig. 2(d) which was obtained by annealing a buried P layer (with no H present) to 600 °C. While a sufficient number of P atoms have diffused to the surface to yield a high density of Si-P heterodimers,

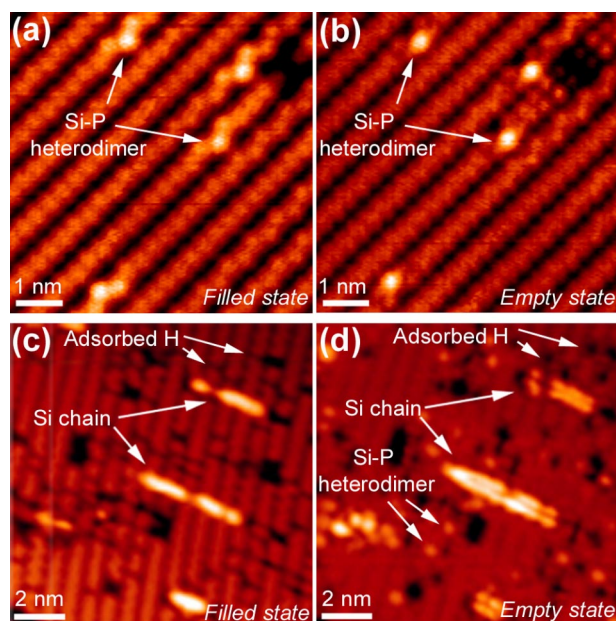


FIG. 4. (a) Filled state and (b) empty state STM images of a Si(001) surface after dosing with 0.002 L of PH_3 and annealing to 500 °C. (c) Filled state and (d) empty state STM images of a Si(001) surface after dosing with 0.013 L of PH_3 and annealing to 350 °C. Tunneling conditions were (a) and (c) -1.6 V and 0.13 nA; (b) and (d) $+1.2$ V and 0.13 nA.

the surface is free of H. In this image the Si-P heterodimers are clearly visible, which is consistent with the observation that it is the high density of monohydrides on the surface that inhibits the STM observation of Si-P heterodimers.

Our final anneal of the PH_3 /Si(001) 0.002 L dosed surface (not shown) was performed at 700 °C and resulted in the disappearance of the Si-P heterodimers. By comparing our results with that from the annealing of a saturation dosed PH_3 /Si(001) surface,¹⁰ we conclude that the P atoms have desorbed from the surface, leaving only features inherent to the clean Si(001) surface.

D. Identification of the Si-P heterodimer using empty state imaging

In the preceding section we showed that for high coverages of monohydrides, the identification of Si-P heterodimers in filled state images was not possible. However, we will show that we can clearly identify Si-P heterodimers, at any coverage, using empty state imaging. Figure 4 shows (a) filled- and (b) empty-state images of the same area of a Si(001) sample after a 0.002 L PH_3 dose and a 500 °C anneal. We have chosen a low PH_3 dose and an anneal temperature sufficiently high such that ejected Si dimer chains and monohydrides have been completely removed from the surface, so that we can image Si-P heterodimers in isolation from other features. Figure 4(b) shows that in empty state images the Si-P heterodimer appears as a bright circular protrusion on one side of a dimer row with a slight depression on the other side of the row. The height of the protrusion, measured with respect to the surface dimers, varies linearly with bias between 0.50 ± 0.05 Å at $+0.8$ V and 0.10

$\pm 0.02 \text{ \AA}$ at $+2.0 \text{ V}$. Dimer buckling in empty state images of Si(001) is not observed for sample biases greater than $+0.8 \text{ V}$.²³

With the Si-P heterodimer well characterized in both filled and empty state imaging we can now also interpret images obtained after higher PH₃ doses but a lower anneal temperature, where H is still present. Figures 4(c) and 4(d) are dual bias images of a Si(001) surface that has been dosed with 0.013 L of PH₃ and annealed to 350 °C. The filled state image shows a number of Si chains and monohydrides but no Si-P heterodimers are observed. We see that the ejected Si chains have a characteristic minima running lengthways along their center in the empty state image, as expected.^{14,17} However, despite the difficulty in resolving features due to the extreme brightness of the ejected Si, in the empty state image we still see circular protrusions characteristic of the Si-P heterodimers, as well as the Si chains and the monohydrides. This ability to identify incorporated P atoms regardless of the density of other features on the surface is a useful tool for locating P atoms in Si, towards the goal of realizing atomic-scale devices.^{1,3}

IV. CONCLUSION

We have used STM and AES to study the incorporation of P into Si(001) by dosing with small quantities of PH₃ and

annealing. The Si-P heterodimer appears as a zigzag feature when imaged in filled state STM images due to the static dimer buckling induced by the heterodimer. The Si-P assignment was confirmed from the direct correlation between the number of zigzag features imaged by STM and the intensity of the P 120 eV Auger peak. The presence of moderate numbers of monohydrides on the surface removes the buckling induced by the Si-P heterodimer and renders the heterodimer invisible in filled state images. We characterized the Si-P heterodimer using empty state imaging and show that in the empty state the Si-P heterodimers are visible even with a high coverage of monohydrides. The demonstration of the incorporation of individual P atoms into the Si(001) surface, and subsequent characterization using STM, is an important step towards fabrication of nanoscale electronic devices based on controlled atomic-scale doping of Si.

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