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# Studying the lateral composition in Ge quantum dots on Si(0 0 1) by conductive atomic force microscopy

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**Abstract**

Conductive atomic force microscopy (C-AFM) has been employed to investigate the distribution of the lateral composition in Ge quantum dots (QDs) grown by molecular beam epitaxy on p-type Si(0 0 1) substrate. Since the different conductivity of the components (Ge and Si) in the Ge QDs results in different current signals, it is then possible to obtain the information of composition distribution from the current images. We have investigated two types of samples grown at 550 °C and 640 °C, respectively, and found that the conductance distribution of these two types of QDs were significantly different. This difference can be attributed to the different degrees of Si alloying into the Ge QDs at different growth temperatures. Our results demonstrate that the dome-shaped QDs grown at the higher temperature are Si–Ge alloys with Si composition >35% at most part of the QD, while the QDs with the same shape grown at the lower temperature show high Ge distribution (>65%) in the whole dot, which are supported by the selective etching experiments.


*Keywords:* Conductive atomic force microscopy (C-AFM); Quantum dots (QDs); Conductance; Composition profile; Ge; Si

## 1. Introduction

Recently, self-assembled Ge quantum dots have attracted intense interests and been widely studied for promising applications in low-dimensional optical and electronic devices, such as single-electron transistors, far-infrared detectors and quantum dot lasers. A full understanding of the composition profile of self-assembled semiconductor QDs is of fundamental importance for applications because predictions about any optical and electronic properties in such QD-based devices are predicted reliably only if the correct material distribution is known. Several independent studies on the composition of self-assembled Ge QDs by selective etching, X-ray diffraction, X-ray photoemission spectroscopy, cross-section TEM have been reported [1–4]. However, these techniques

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either give area-averaged information containing an ensemble of dots or destroy the samples. Therefore, approaches based on scanning probe techniques are being considered.

In this work, we employed conductive atomic force microscopy (C-AFM) to study the composition distribution in Ge quantum dots fabricated on silicon substrate by analyzing their local electrical properties. C-AFM is a promising technique capable of performing local conductance measurements over a thin film surface, and it has been already used for investigating local electrical properties on semiconductors, film dislocation, carrier profiling and charge injection [5–8]. Since the conductivity of Ge is higher than that of Si, thus it is possible to employ C-AFM to image the distribution of the conductivity in a single quantum dot. Hence we can obtain the composition profile. Nominally pure Ge QDs are substantially intermixed with Si when the growth temperature is above 550 °C and the mixture degree increases with the temperature, therefore C-AFM is a powerful technique to study the Ge and Si diffusion in a single quantum dot as a function of growth temperature. In this paper, C-AFM characterizations of two kinds of samples grown at different temperatures are compared, and the results are confirmed by selective etching experiments.

## 2. Experimental

The self-assembled Ge QDs studied in our experiments were prepared by solid source molecular beam epitaxy (Riber Eva-32) on p-type Si(001) substrates with resistivity of 1–10 Ω cm. Before deposition, the substrates were chemically cleaned using the Shiraki method [9], and the resulting protective oxide layer was removed by heating at 1000 °C for ten minutes in the growth chamber. Then the substrate temperature was lowered to 650 °C, and 50 nm Si buffer layer was grown. Sample A was prepared by depositing 1.7 nm Ge at 550 °C, and then the sample was immediately cooled down to room temperature. For sample B, 0.85 nm Ge layer was deposited at 640 °C, and the sample was held at 640 °C for 5 min before cooling down [10,11].

Sample morphology and current images of the samples were measured by C-AFM. A commercial ambient AFM (Solver P47, NT-MDT) system was used for the AFM experiments. In the C-AFM measurements, Pt or TiN coated Si tips scanned over the sample surfaces in the contact mode (constant force) with a bias voltage. Positive bias voltages were applied to the substrate while the tip was grounded. The current between the tip and sample was measured simultaneously with the surface topography imaging, allowing direct correlation of structural features with their electrical characteristics. Before the C-AFM experiments, samples were treated at room temperature by diluted hydrofluoric acid (original 40% and dilution ratio 1:10) to remove the oxide layer. The experiments were performed in nitrogen gas to protect the samples from oxidation. For relative etching experiments, the Ge QDs were wet chemically etched in a hydrogen peroxide solution (30%), and the morphology and current images of the dots after etching were also analyzed by C-AFM for comparison.

## 3. Results and discussion

Fig. 1(a) and (b) shows the topographic and current images of Ge QDs (sample A, 1.7 nm Ge deposition at 550 °C) with a bias voltage of 1.0 V, respectively. The cursor profiles along the solid lines as marked in (a) and (b) are shown in Fig. 1(c). Uniform and coherent Ge QDs with two typical sizes are observed in the topographic image: the larger dots are dome shape with an average diameter of 100 nm and an average height of 12 nm, and the smaller dots are mound-shaped with an average height of 1.0 nm. To confirm the observed QDs sizes, the sample was also measured in contact mode with non-coated AFM tips, which are sharper than the coated ones, and identical results were obtained. In this paper, we mainly concern the dome-shaped QDs. Current image obtained at 1.0 V is shown in Fig. 1(b), with the cross-section profile along the solid line given in Fig. 1(c). Since Ge is readily oxidized in air, the absolute value of the measured current is weakened due to the oxidation. In our experiments, we found the distribution of the current in the

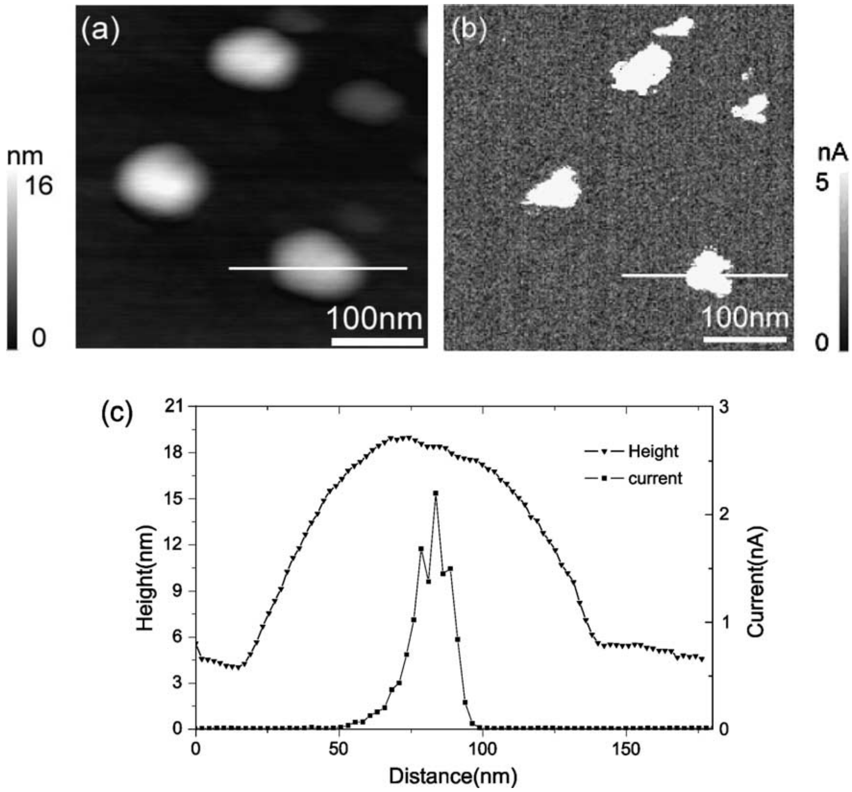


Fig. 1. Topographic (a) and current (b) images of Ge QDs grown at 550 °C. Both images were obtained simultaneously at a sample bias voltage of 1.0 V and the scan size was 400 nm × 400 nm. (c) Respective cross-sectional profiles along the solid lines marked in (a) and (b).

QDs was well repeated, while the absolute values of the current were not reproducible. Therefore, in this paper we concerned the distribution of the current, rather than the absolute value of the current. The local conductance distribution of a single QD is revealed. For all QDs shown in Fig. 1(b), the conductance at the center of the QD is higher than that at its periphery. The resistance of the wetting layer is found to be much higher than that of the quantum dot, because the current at the wetting layer is too low to be detected.

Our results are similar to the results reported for self-assembled InAs QDs quantum structure system by Tanaka et al. [12], in which the differences in the conductance were attributed to the local modification of surface band bending associated with the surface states in the QDs and wetting layer. No composition distribution was discussed in their work. For SiGe QDs, Si is easily alloyed into the Ge QDs, therefore the composition distribution of the SiGe QD should be considered in our experiments. The composition profiles of Ge QDs on Si have already been studied by other techniques, such as X-ray diffraction, transmission electron microscopy [2,4]. Most results support the existence of a distinct SiGe vertical composition variation, with a Si-rich core covered by a Ge-rich shell in a dome-shaped QD. In our case, as confirmed by the following etching experiments, the Si-rich core is small due to the low deposition temperature. Thus the composition distribution induced current difference can be ignored here. The differences in the conductance can also be attributed to modification of Schottky barrier height. In current measurements, the sample surface is positively biased. Positive charge accumulation occurs on the surface of Ge dot, which leads to the lowering of Schottky barrier height, and the effect of surface potential lowering would be larger at the center of the QD than at its periphery [13]. As a result, the measured current is higher at the center of the QD than at its periphery.

Noticeable difference is observed in the conductance characteristics of sample B (0.85 nm Ge deposition at 640 °C). Topographic and current images of Ge QDs obtained simultaneously with a bias voltage of 1.4 V are shown in Fig. 2(a) and (b), respectively. It can be observed that the QDs are relatively uniform domes in size and height, with an average diameter of 70 nm and an average height of 18 nm. The current profile along the solid line as marked in Fig. 2(b) is shown in Fig. 2(c), which gives the conductance distribution of a single QD. The clearly observed local contrast in the current image is surprising: periphery of the QD is more conductive than its center. This result is significantly different from that of sample A as shown in Fig. 1, and also different from the reported results for InAs QDs [12]. In literature [14], similar results were reported for $TiSi_2$ on Si, where some $TiSi_2$ islands showed higher conductance at the edge than that at the center. It was explained that the electric field would be bunched by the formed facets at the edges, leading toward a lowering of the Schottky barrier height at the edge. In our case, since the QDs with the same shape (samples A and B) show different conductance distribution, facets induced Schottky barrier lowering is not the main factor determining the conductance distribution, and we intend to attribute the different conductance distribution to the different diffusion levels of Si into the Ge dots.

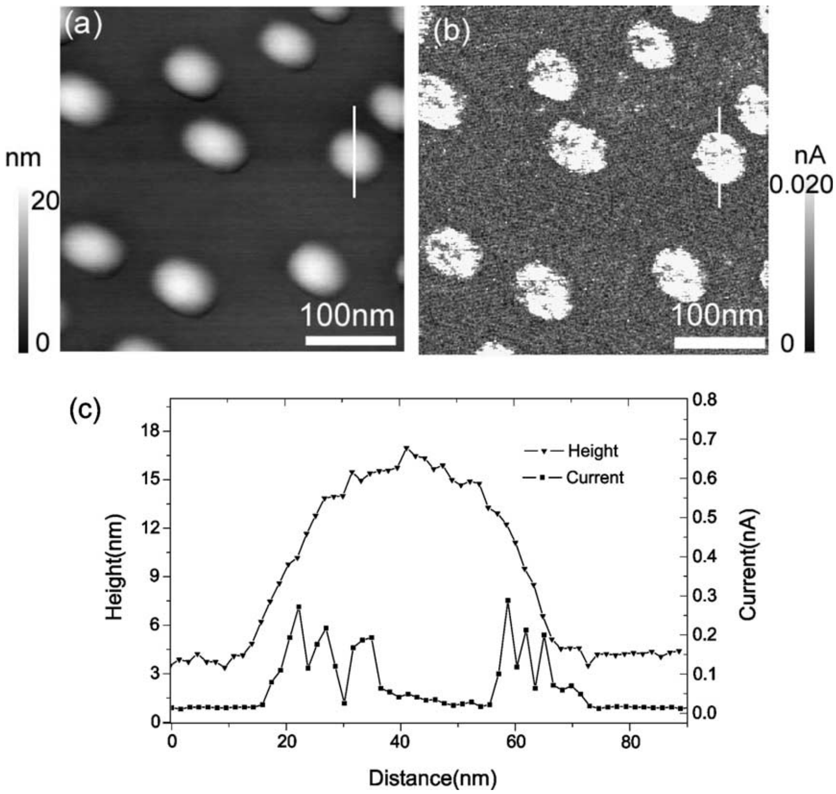


Fig. 2. Topographic (a) and current (b) images of Ge QDs grown at 640 °C. Both images were obtained simultaneously at a sample bias voltage of 1.4 V and the scan size was 400 nm × 400 nm. (c) Respective cross-sectional profiles along the solid lines marked in (a) and (b).

It is known that nominally pure Ge QDs are substantially intermixed with Si when the growth temperature is above 550 °C [1] and the conductivity of Ge is higher than that of Si. Sample A was deposited at 550 °C followed by an immediate cooling down to room temperature after Ge layer deposition, thus only a little amount of Si is alloyed into the Ge dots. On the contrary, sample B was deposited at 640 °C and held at this temperature for 5 min before cooling down, thus a large amount of silicon was alloyed into the quantum dot, which largely increased the resistance of the dot. The total resistance measured includes the contact resistance between the tip and the dot ($R_C$), the resistance of the dot itself ($R_D$), and the resistance between the dot base and the metal contact tap on the surface ($R_S$). $R_S$ is much smaller than the other two because of the large contact area, and will not be taken into account. The contact resistance $R_C$ is related to the Scottky barrier between the tip and the surface, and it is dominated for sample A. For sample B, the dot resistance $R_D$ is largely increased since Si is heavily alloyed into the dot, and it becomes to dominate the total resistance. Because $R_D$ is proportional to its current length, i.e., dot height, $R_D$ has smaller value at its periphery than at its center, which results in the current distribution of sample B as shown in Fig. 2(b).

In order to confirm the above interpretation, the composition distribution of Ge in the dots was also investigated by selective wet chemical etching experiments. Sample A and B were etched by using 30% $H_2O_2$ for 30 min at room

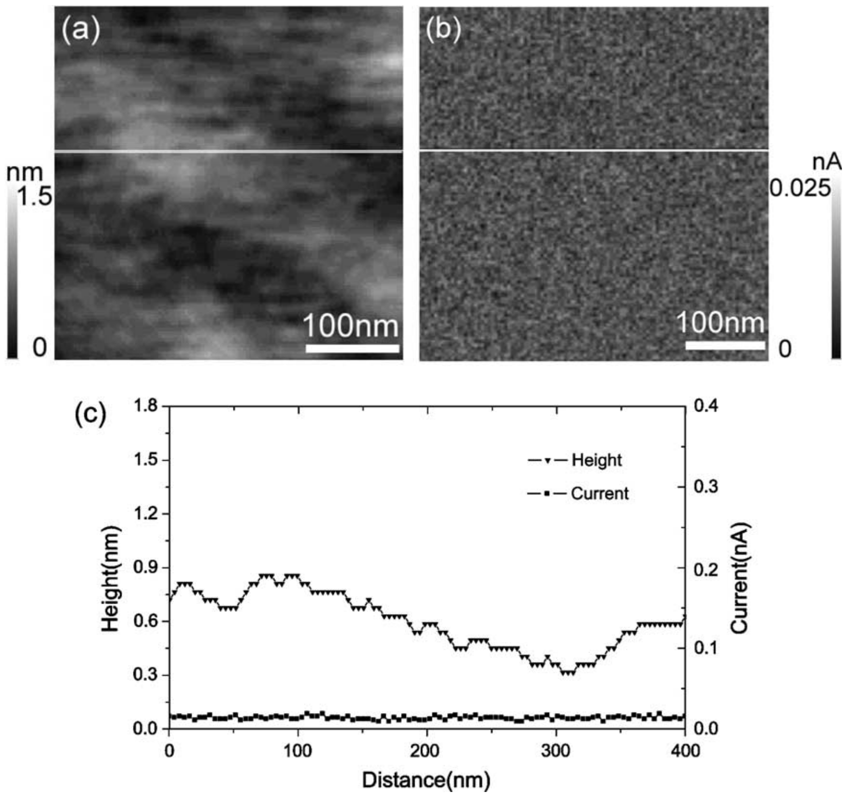


Fig. 3. Topographic (a) and current (b) images of Ge QDs grown at 550 °C after etching in 30% $H_2O_2$ solution for 30 min. Both images were obtained simultaneously at a sample bias voltage of 1.0 V and the scan size was 400 nm × 400 nm. (c) Respective cross-sectional profiles along the solid lines marked in (a) and (b).

temperature. This etchant can selectively remove GeSi material with Ge composition higher than 65 ± 5% [1], leaving GeSi material with Ge composition less than 65% on the surface. As shown in Fig. 3(a), the dots grown at 550 °C (sample A) are completely etched away, leaving a flat surface, which indicates that Ge composition in dots is higher than 65%. However, for sample B (grown at 640 °C), as shown in Fig. 4(a), only a small part of the dot is etched away and much more residual matter of the QDs is left on the surface, with the dot shape still remaining. By comparing the average dot size before and after etching, both the average height and diameter of the QD were a little reduced after etching, indicating the thin cover layer of the QD with Ge > 65% being removed by etching. It suggests that the most part of the QDs of sample B contains Si higher than 35% and this high Si concentration is definitely due to Si atoms mixing into Ge dots at the growth temperature of 640 °C. Similar phenomenon was observed by Denker et al. [1] using selective etching and they found that the dome islands exhibit an anisotropic shape with only one etched corner. It was explained that the asymmetries during the transition from pyramid to dome caused a very asymmetric Ge profile. Also, current images were measured on the two etched samples, as shown in Figs. 3(b) and 4(b), respectively. No current signals could be observed in our current detecting limitation (~10 pA). The cross-sections of the current on the etched surfaces are shown in Figs. 3(c) and 4(c), and no current can be detected. It is because that the residual matter on the surface

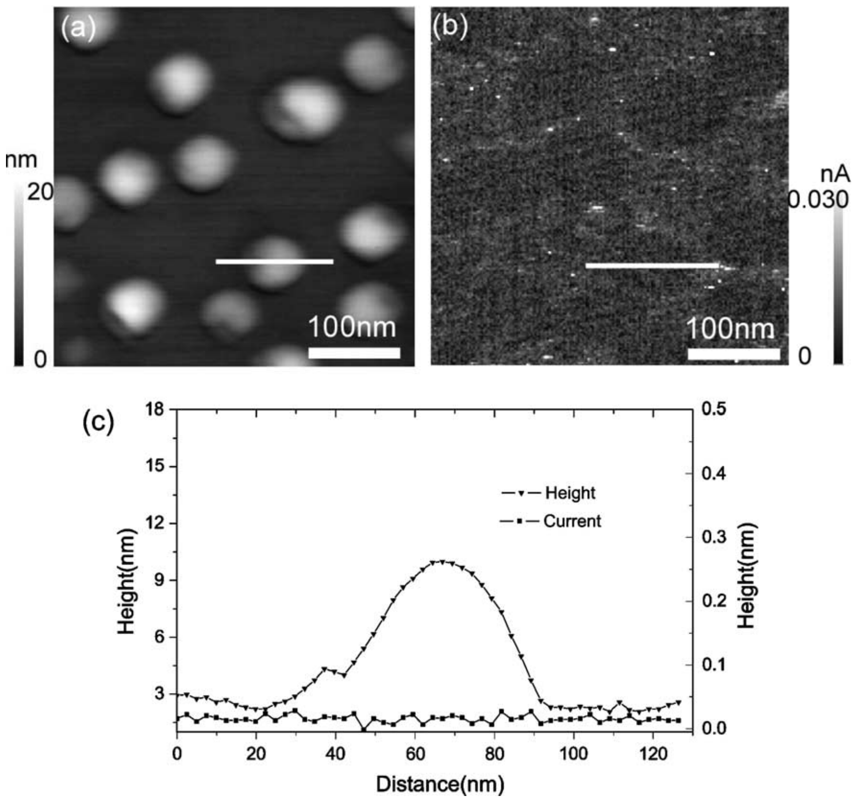


Fig. 4. Topographic (a) and current (b) images of Ge QDs grown at 640 °C after etching in 30% $H_2O_2$ solution for 30 min. Both images were obtained simultaneously at a sample bias voltage of 1.9 V and the scan size was 400 nm × 400 nm. (c) Respective cross-sectional profiles along the solid lines marked in (a) and (b).

contains Si larger than 35%, resulting in a low current which is beyond the detect limitation.

From the above, the composition profiles of SiGe QDs grown at 550 °C and 640 °C are obtained, respectively. Though the dots are both dome-shaped in morphology, their composition distribution is different which is caused by the different degree of Si and Ge intermixing at different growth temperatures, and this interpretation is well supported by the results of etching experiments.

## 4. Conclusion

In conclusion, we have used C-AFM technique to investigate the lateral composition distribution in Ge quantum dots (QDs) grown at 550 °C and 640 °C on Si(001) substrate, respectively. Significant difference in local conductance distribution has been observed on these two types of QDs. The QD grown at 550 °C exhibits higher conductance at the center than at its periphery, while the QD grown at 640 °C shows that the periphery is more conductive than its center. This difference is attributed the different concentrations of Ge/Si components in these two types of Ge QDs. By combining the results of etching experiments, it can be obtained that Ge QDs grown at 640 °C have Si concentration larger than 35% at most part of the dots, while the QDs grown at 550 °C contain high Ge concentration (>65%) in the whole dots. Such observations provide that C-AFM is a promising technique to study the material composition distribution inside nano-structures conveniently without destroying the sample surface.

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