Chapter 5 SiO_2/Si(111) Height Error by Atomic Force Microscopy

5.1 Introduction

Semiconductor surfaces and interfaces are very important for silicon devices as well as fundamental physics. When we understand more of their atomic and electronic structure, we can apply them more and more. As an example, SiO_2 is indispensable for the fabrication of Si-based devices. Silicon dioxide (SiO_2) films are an excellent insulating material because the interface between the SiO_2 film and the Si substrate are abrupt and stable. The thermally grown ultrathin SiO_2 films have been studied extensively. As the device miniaturization trends continue, SiO_2 films used as gate dielectric layers with thicknesses less than 1 nm may be needed within a decade [57,58,59]. The SiO_2 films are also useful as masks for etching and selective growth. Therefore, nanometer-scale patterning onto SiO_2 layers will be an important step towards the development of advanced quantum-effect devices.

Although several detailed interface structures between the SiO_2 layer and the bulk Si have been proposed, there still exist open questions such as local hot spots and trapping charges [60,61]. In our Lab., NC-AFM can be operated in the STM mode, contact mode, and noncontact mode by using different scanning probes and different control circuits. STM images can achieve true atomic resolution and the others can achieve nanometer resolution. We grew the ultrathin SiO_2 films *in situ* and attempted to gain more understanding on the structure and physical properties by combining all the information obtained by these real-space microscopic techniques.

5.2 Experiment

Ultrathin SiO_2 films react with air easily. Ultrathin SiO_2 films and a few monolayers can only be prepared in UHV system *in situ*. Our UHV chamber has a base pressure of 1×10^{-10} torr and incorporates an UHV AFM/STM based on a tube piezoelectric scanner. To obtain the low pressure, various pumps such as dry pump, turbo pump, ion pump, and baking process are needed. To produce ultrathin SiO_2 layers, we follow the procedure developed by W. Jun *et al.* [62].

Our first topic is to grow the SiO_2 on the Si(111) surface in a layer by layer fashion. The samples were loaded into the UHV chamber, and degassed to remove the native oxide layers. After the samples were flashed at 1200 ℃ by directly passing current through the sample, the clean Si(111)-(7×7) surface were obtained as shown in Fig. 5.1(a). In the STM image we can see an atomic step between the two terraces (labeled A and B). Atoms are clearly seen the zoom-in image (Fig. 5.1(b)). The step height between the two terraces measures here about 0.31 nm from the line-profile shown in Fig. 5.1(c).

To grow a SiO_2 film on the clean Si(111)-(7×7) surface, the vacuum chamber was backed filled with high purity dry oxygen to a pressure of 2×10^{-5} torr while the sample was kept at 775 ℃ for 5 mins. We typically annealed the sample at 800 ℃ for 5 ~ 10 mins to create the SiO_2 voids via SiO desorption. In this experiment, the void's size of the SiO desorption is about $100\times100\ nm^2$. We want to avoid the SiC structure appearing on the surface in bigger void's size. The Figure 5.2(a) and 5.2(b) display the schematics for the SiO_2/Si(111) before and after annealing. The step height is about 0.7 nm between Si(111) and SiO_2 layer. The SiO desorption proceeds via a pathway,

$$Si(s) + \frac{1}{2}O_2\,(g) \rightarrow SiO\,(g)$$

with s and g is in the brackets denoting solid and gas phase, respectively.

Heiji Watanabe *et al.* presented an oxide interface model between Si(111) and SiO_2 layer. This model can be calculated SiO_2 thickness of one layer [63]:

the volume of Si atom per mole: $\frac{28.09}{2.33} = 12.06\ cm^3$,

the weight of SiO_2 per mole: $60.08\ g$,

the density of SiO_2: $2.21\ g/cm^3$,

the volume of SiO_2 per mole: $\frac{60.08}{2.21} = 27.19\ cm^3$,

$Si(s) + \frac{1}{2} O_2\ (g) \rightarrow SiO\ (g)$ ，

$$\frac{\text{The volume of Si atom per mole}}{\text{The volume of SiO}_2\text{ per mole}} = 2.25,$$

$$\text{One bilayer of } SiO_2 \text{ thickness} = 2.25 \times \text{One bilayer of Si (0.31 nm)}$$
$$\cong 2.25 \times 0.31 \cong 0.7 \text{ nm.}$$

In our experiment, STM and NC-AFM measurements were performed in ultra-high vacuum chamber with a based pressure of 8×10^{-11} torr and equipped with a commercial variable-temperature STM/NC-AFM system. We used commercial heavily doped monolithic Si cantilevers with tip radius of less than 7 nm. The typical force constant k is 42 N/m and free resonance frequency f_0, 260 KHz. Conductive tips have an additional 25-nm thick double layer of Chromium and PtIr5. All images in this work were taken with a cantilever oscillation amplitude (A) of around 16 nm and a frequency shift Δf = -30 Hz.

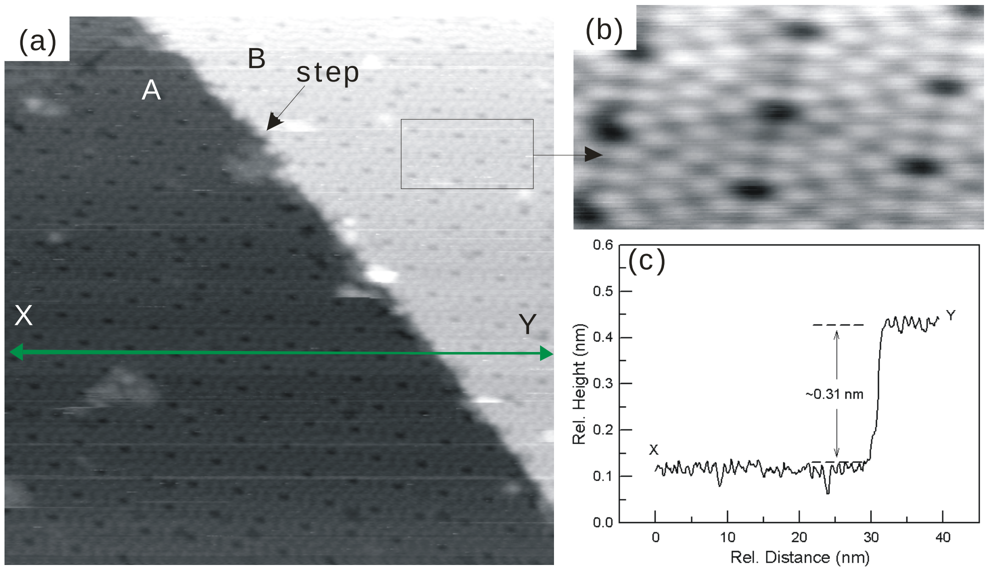


Fig. 5.1 (a). STM image for the Si(111)-(7×7) surface. (b) Zoom-in image from (a). (c) The line-profile between X-Y in (a). The measurement step-height is about 0.31 nm.

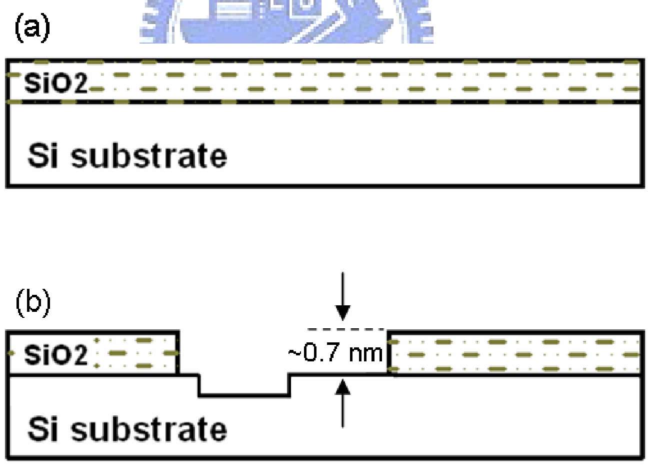


Fig. 5.2 (a) The schematic showing a SiO_2 film was created by thermal oxidation. (b) Voids can be created by annealing ~800 ℃ for 5 ~ 10 mins. The SiO_2 thickness is about 0.7 nm.

5.3 Results and Discussion

5.3.1 AFM images and line-profiles

(a) Si cantilever

In this experiment, the Si cantilever is used to observe the SiO_2/Si(111) as shown in Fig. 5.7. The images were obtained in AFM mode (fig. 5.7(a-e)) and STM mode (fig. 5.7(f)). When the compensated bias was applied, the clear image and true were obtained as shown in Fig 5.7(a), and the Si(111) step and Si(111)-SiO_2 can be obtain. The line-profiles are shown in Fig. 5.7(h). The tip bias was applied with -6, -3, -0.5, 3, 6V, and STM image for (a), (b), (c), (d), (e), and (f), respectively. The Si cantilever is a sharp tip, and then the height variation of voids had a broad bias range. The blurred image can be obtained on positive tip bias. The illustration was shown the Si(111)-7×7 and SiO_2 layer to correspond with the line-profiles as shown in Fig. 5.7(g).

STM mode can not distinguish height difference of the Si(111) and the SiO_2 layer. The SiO_2 layer is an insulated layer for tunneling current, so the feedback system that detects the tunneling current will not active. The Si(111) the SiO_2 layer almost have the same height as shown in Fig. 5.3 (g). The SiO_2 layer is only obtained some roughness in the line-profile.

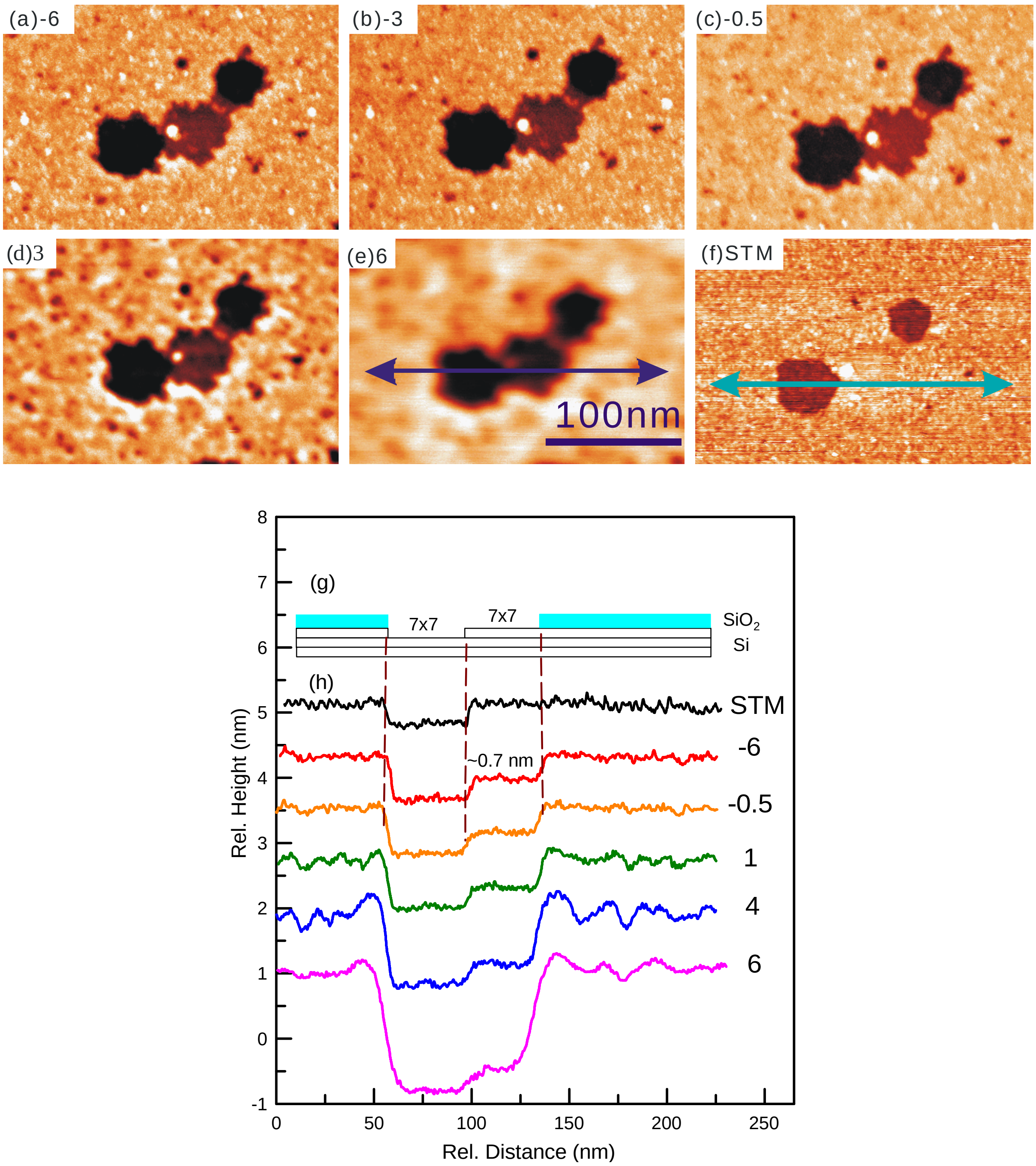


Fig 5.3 NC-AFM topography image (250×250 nm^2) of the SiO_2 films on the Si(111) surface using sharp Si tip. The AFM images were obtained using tip bias with -6, -3, -0.5, 3, and 6 V correspond to (a), (b), (c), (d), and (e), respectively. STM image was obtained in the same area as shown in the (f). (g) is the illustration for the AFM image and correspond to line-profile in (h). (h) shows the line-profiles taken from (a)-(f).

Figure 5.4 showed the relative step height that measured from line-profile for the different bias. The SiO_2 thickness was 0.7 nm from the calculation, and plotted using a dash-line. The step height can be obtained 0.7 nm between -6 V and 0 V, and got the more heights by increasing tip bias. The blurred image and error height can be acquired with the increasing bias, and the AFM image was shown in Figs. 5.3 (a-e). The height variations are caused from the electrostatic force. The electrostatic force is discussed in the Chapter 3.

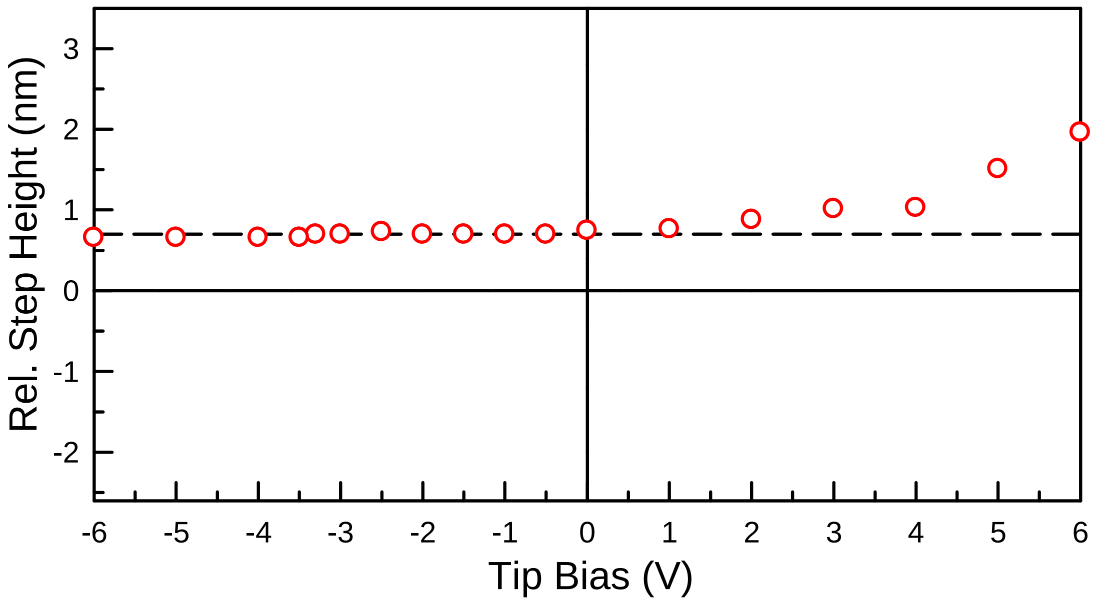


Fig. 5.4 The relative step height is as function of tip bias for Si-tip. For Si-tip, there are different step heights were measured from the different tip bias. The dash-line shows the calculation of SiO_2 thickness at 0.7 nm.

(b) Sharp PtIr5 cantilever

AFM was employed to get the topography images for the SiO_2/Si(111) surface using PtIr5 cantilever. The results are shown in Figs. 5.5(a-e). These images were taken sequentially in the non-contact mode on the same area. They were obtained with tip bias at different voltage. We choose Fig. 5.5(a) for example to interpret the meaning of the image. We applied -4 V on the tip, while scanning. In Fig. 5.5(a), we can see invert image that the light area is Si(111) and other is SiO_2 layer . A line-profile across the image is plotted in Fig. 5.5(g). The height variation of all measures is shown in Figs. 5.5(a-f). The true depth of the voids measures about 0.7 nm form bottom of the void to the top of the SiO_2 layer.

The line-profile of the same position from each image (Figs. 5.5(b-e)) was plotted in Fig. 5.5(g). Figure 5.5(g) show the different void heights with different tip bias. The different void heights show that tip bias will affect the topography image. The depth of the same voids taken from Fig. 5.5(g) were plotted them in Fig. 5.6. Figure 5.6 shows the step height with the positive tip-bias has larger variations than the negative tip-bias.

The Voids size is about 100×100 nm^2. Apply tip bias more and more, then the AFM images are blurred gradually. The PtIr5 usually has a bigger tip radius than the Si-tip, and the effect of the electrostatic force is larger than the Si-tip. The variations of the step heights have changes larger than the Si-cantilever.

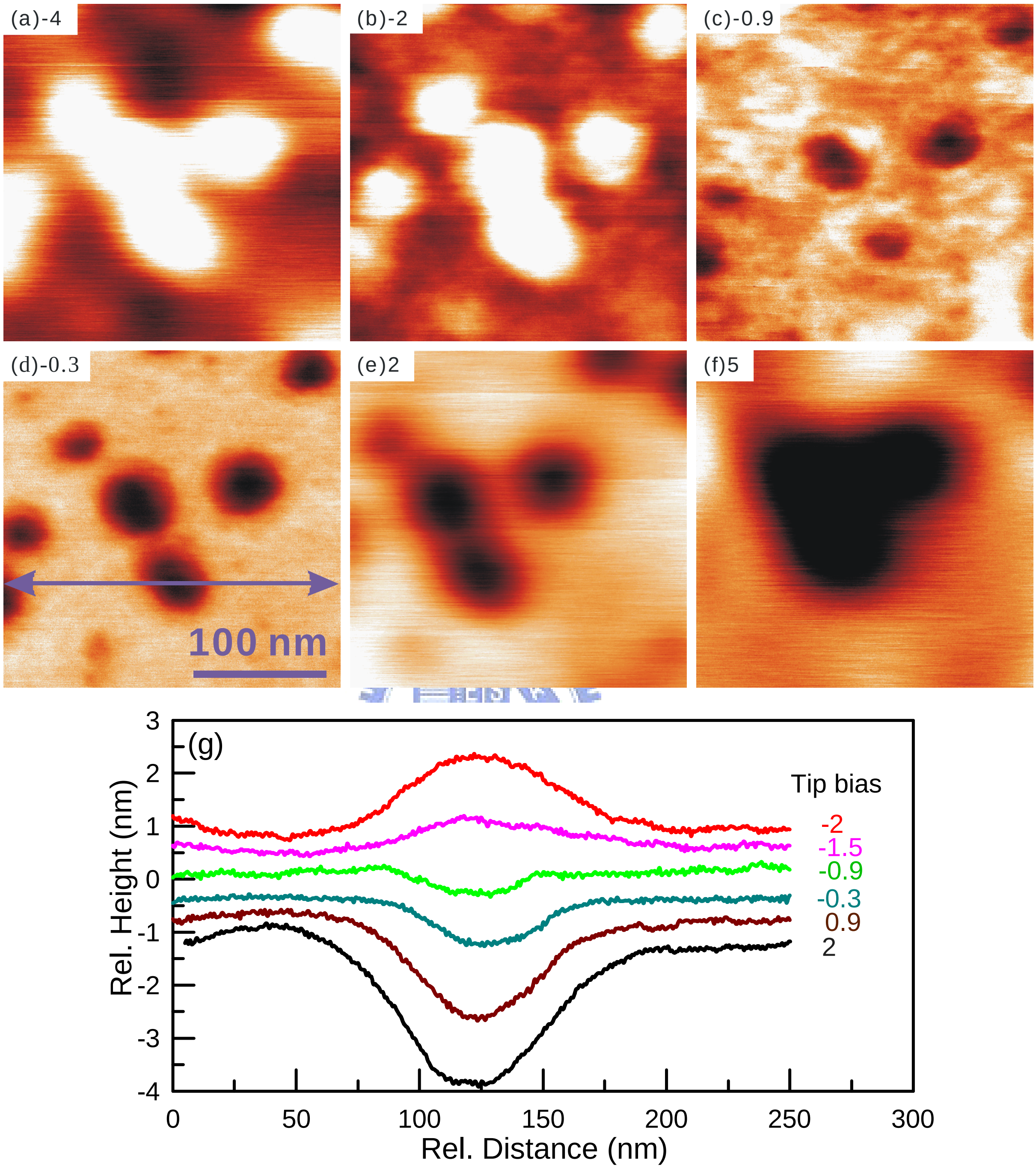


Fig. 5.5 NC-AFM images (250×250 nm^2) were obtained by using sharp PtIr5 tip. The tip bias was applied with -4, -2, -0.9, -0.3, 2, and 5 V for (a), (b), (c), (d), (e), and (f), respectively. (g) shows the line-profiles taken from (a)-(f).

Figure 5.6 showed the step height that got from different bias images. The real step-height was 0.7 nm, and plotted using a dash-line. The real step height can be obtained at ~ -0.45 V that is the compensated value between the tip and the sample. The apparent variations of the step height are far away the compensated bias. The height inversion can be acquired with decreasing tip bias, and the inversion AFM images were shown in Fig. 5.5 (a), and (b).

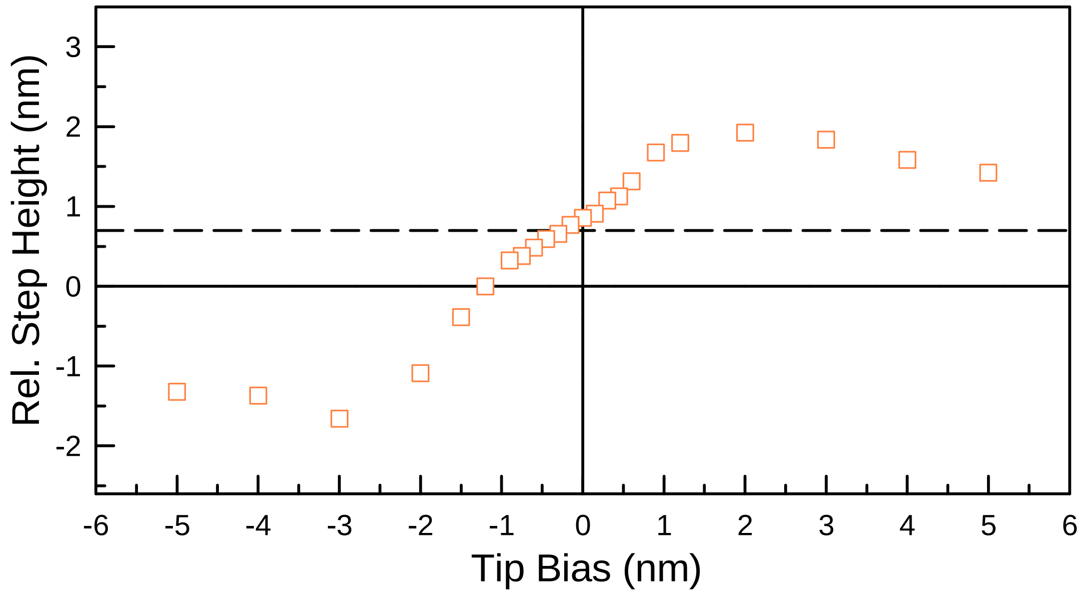


Fig 5.6 The relative step height is as function of tip bias for the sharp PtIr5-tip. For the Sharp PtIr5-tip, there are different void's heights were measured from the different tip bias. The height inversion can be acquired with negative tip bias. The dash-line shows the calculation of SiO_2 thickness at 0.7 nm.

(c) Blunt PtIr5 cantilever

For the blunt PtIr5 tip, the similar experiment was repeated as the sharp PtIr5-cantilever experiment. The AFM images were shown in Figs. 5.7 (a-f). These images were taken sequentially on the same area and were obtained images with the different tip bias. We choose Fig. 5.7(a) for example to interpret the image meanings. We applied -1.5 V on the tip and can obtain the AFM image as shown in Fig. 5.7(a). The image shows inversion, and the light area represents Si(111) substrate and the dark area shows SiO_2 layer. The STM image is obtained in the same area as shown in fig. 5.7(f). A line-profile across the image is plotted in Fig. 5.7(g). The height variation of all measures is shown in Fig. 5.7(g).

The line-profiles of the same position were plotted in Fig. 5.7(g) from Figs. 5.7(b-e). Figure 5.7(g) show the different AFM relative heights with the different tip bias and the STM line-profile. (The line-profile of STM image is three time of the original plot to easily show the height difference.) In the line-profile of STM image, we can get the flat plane on the Si(111) and roughness plane on the SiO_2. The roughness SiO_2 area is cause by the unstable tunneling current because of insulated SiO_2. The different step heights show that tip bias and tip radius will affect the topography image. Figure 5.8 shows that step-height variations and we can obtain higher step height with increasing tip and lower step height with decreasing. The blunt PtIr5 cantilever has large step height variations than the sharp cantilever.

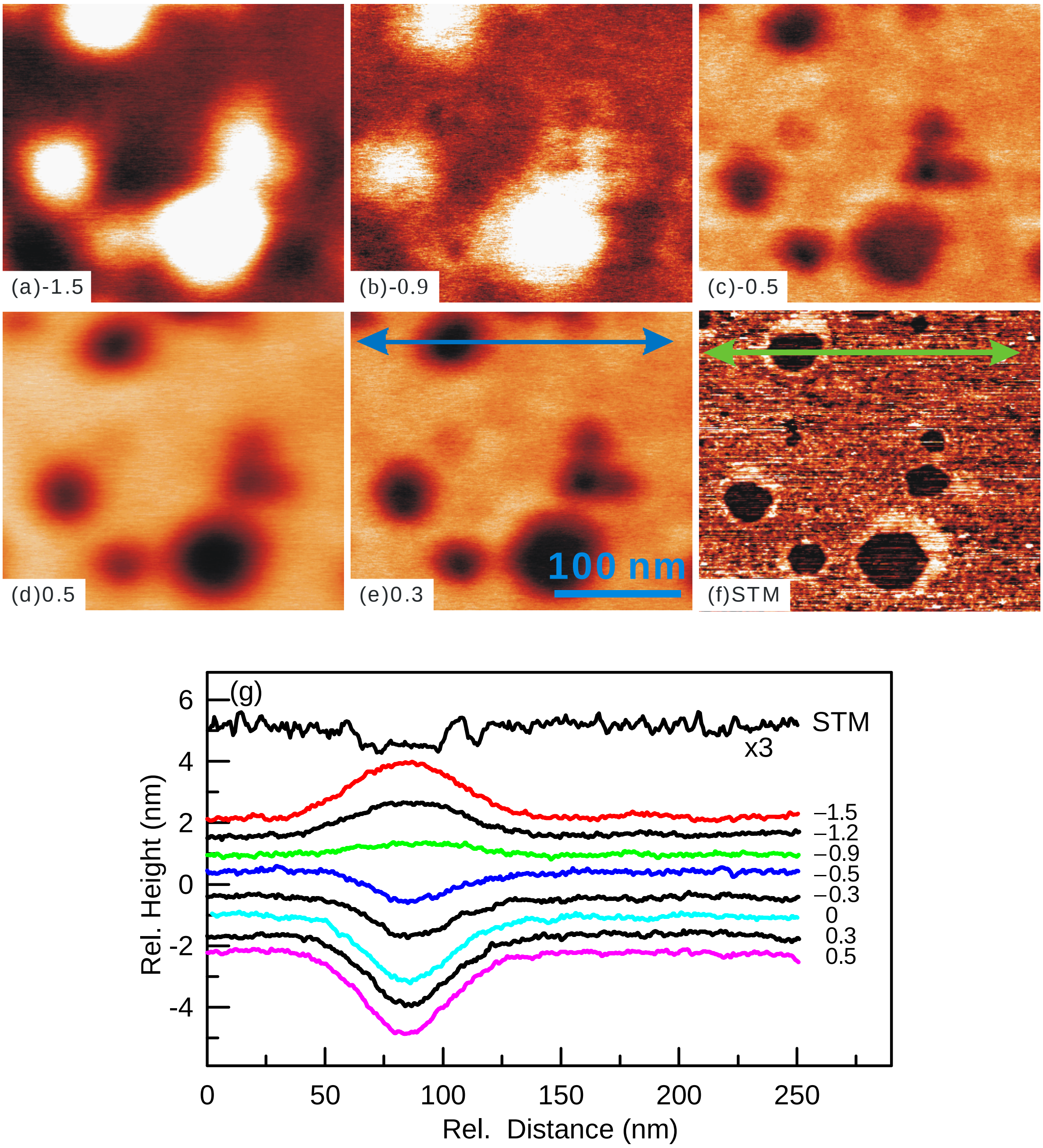


Fig 5.7 NC-AFM topography image (300×300 nm^2) of the SiO_2 films on the Si(111) surface using sharp PtIr5-tip. The tip bias was applied with -1.5, -0.9, -0.5, 0.5, 0.3 V, and STM image for (a), (b), (c), (d), (e), and (f), respectively. (g) shows the line-profiles taken from (a)-(f).

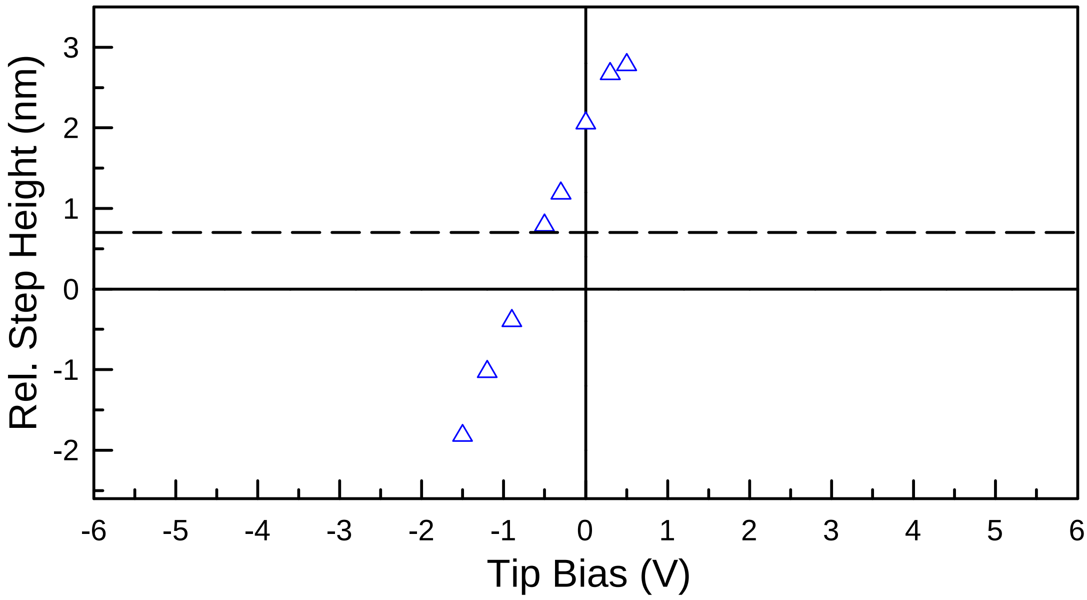


Fig 5.8 The relative step height is as function of tip bias for blunt PtIr5-tip. For Blunt PtIr5-tip, there are different step heights were measured from the different tip bias. The height inversion can be acquired with negative tip bias. The dash-line shows the calculation of SiO_2 thickness at 0.7 nm.

5.3.2 Tip Radius from $D(V_t)$ curve fitting

(a) CPD

The contact potential difference (CPD) is 0.5 V between the Si(111) and the SiO_2 layer by using any PtIr5 cantilever. For example, the PtIr5-tip is measured the $D(V_t)$ curve as shown in the Fig. 5.9. The blunt tip is easier to show the CPD than the sharp tip because the apparent variations in the $D(V_t)$ curve. Also, The CPD can be repeatedly obtained by using different PtIr5-tips, but the curvature variations are dependent on the tip radius. Fig. 5.9 shows the contact potential between PtIr5-tip and Si(111) is -0.1 V ,and the contact potential between PtIr5-tip and SiO_2 layer is -0.6 V. Therefore, the CPD is about 0.5 V between the Si(111) and the SiO_2 layer. In this case, if we want to get a real step height, the tip bias needs to set at the middle of the two contact potentials. Hence, rough 0.35 V is as a compensated bias between the Si(111) and the SiO_2 layer.

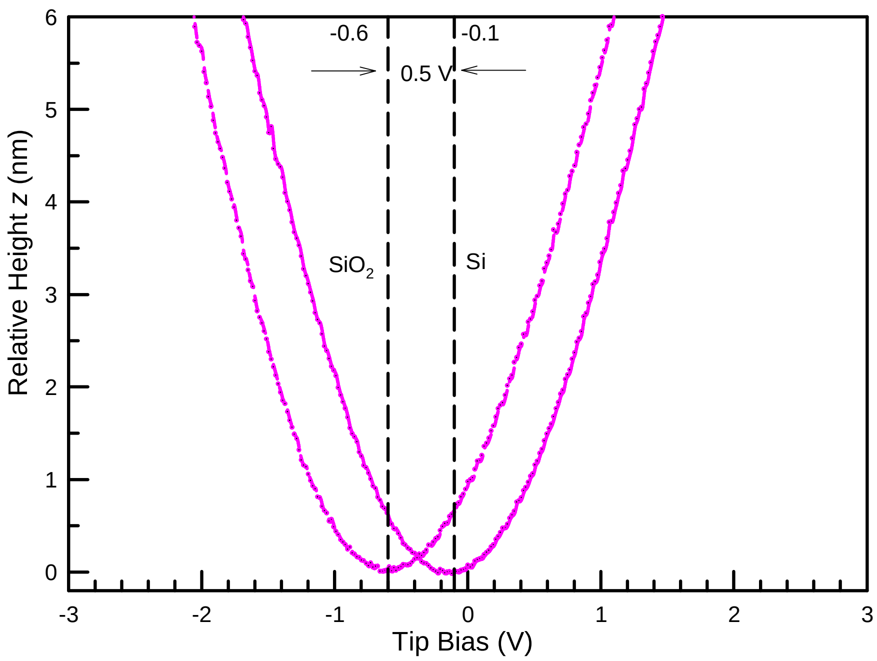
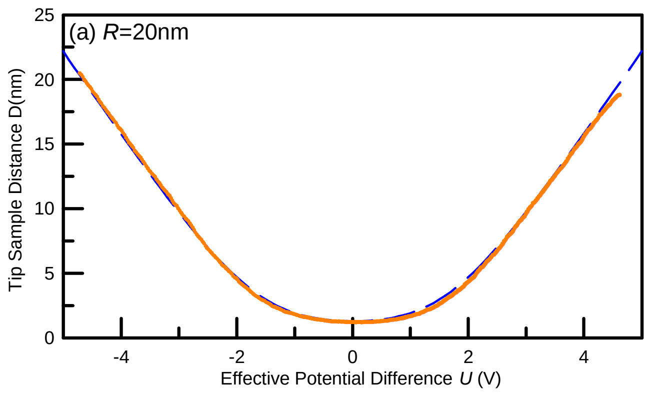


Fig. 5.9 The CPD can be defined from the $D(V_t)$ for the PtIr5-tip. The contact potential between the tip and the Si(111) is -0.1 V and between tip and SiO_2 is -0.6 V. The CPD is 0.5 V between Si and SiO_2 layer using the PtIr5-tip.

(b) The SEM picture

The $D(V_t)$ plot of sharp PtIr5-tip was fitted with the calculation model of weighted integral. The best fitting is R = 20 nm as shown in Fig. 5.10(a). The PtIr5 had shifted the bottom of the curve to the zero bias and showed as relative bias. Hence, we don't need to consider the contact potential and focal on the radius fitting of the cantilever. This method is easy to fit the experimental data with the calculation for different tip radius.

We have the scanning electron microscopy (SEM) image before load the tip into the chamber as shown in Fig. 5.10(b).The SEM image is shown the tip radius is about R = 20 nm which is the nearly same with the calculation of $D(V_t)$ curve.



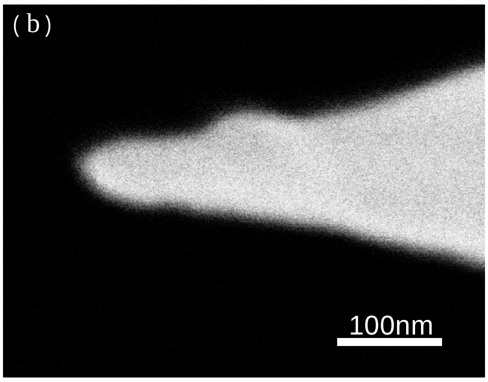


Fig 5.10 (a) The sharp PtIr5-tip fitted with the calculation model of the weighted integral. The fitting radius is R = 20 nm for the sharp PtIr5-tip. (b) It is SEM picture for the sharp PtIr5-tip before scanning. The SEM picture shows the tip radius and consistent with the fitting of the $D(V_t)$.

(c) The $D(V_t)$ curve fitting

The $D(V_t)$ curve of different tips is fitted with the calculation of the $D(V_t)$ as shown in the Fig. 5.11. The dash line is show the $D(V_t)$ calculation for R = 100, 70, 64, 50, 30, 20, 10, and 7 nm with oxide 7 nm, respectively. The experimental $D(V_t)$ plot is shifted the lowest point of the curve to the zero bias as the relative bias. Fig.5.11 shows the $D(V_t)$ variation on the compensated bias.

The fitting bias range is between -1 V and 1 V in the Fig. 5.11. Over ±1 V, the effect of the electrostatic force increase gradually, so tip is far away the sample. When tip is far away the surface, the effect of the electrostatic affects from the Si(111) and the SiO_2 layer simultaneously. It is difficult to exactly distinguish the source of the electrostatic force. The AFM images are also very blurred, and the step height is incorrect with the calculation fitting from the step height versus tip bias which can be shown later. Hence, we discussed the effect of the electrostatic force between -1 V and 1 V. Over ±1 V, the step height is incorrect which is limited by the void size on the sample.

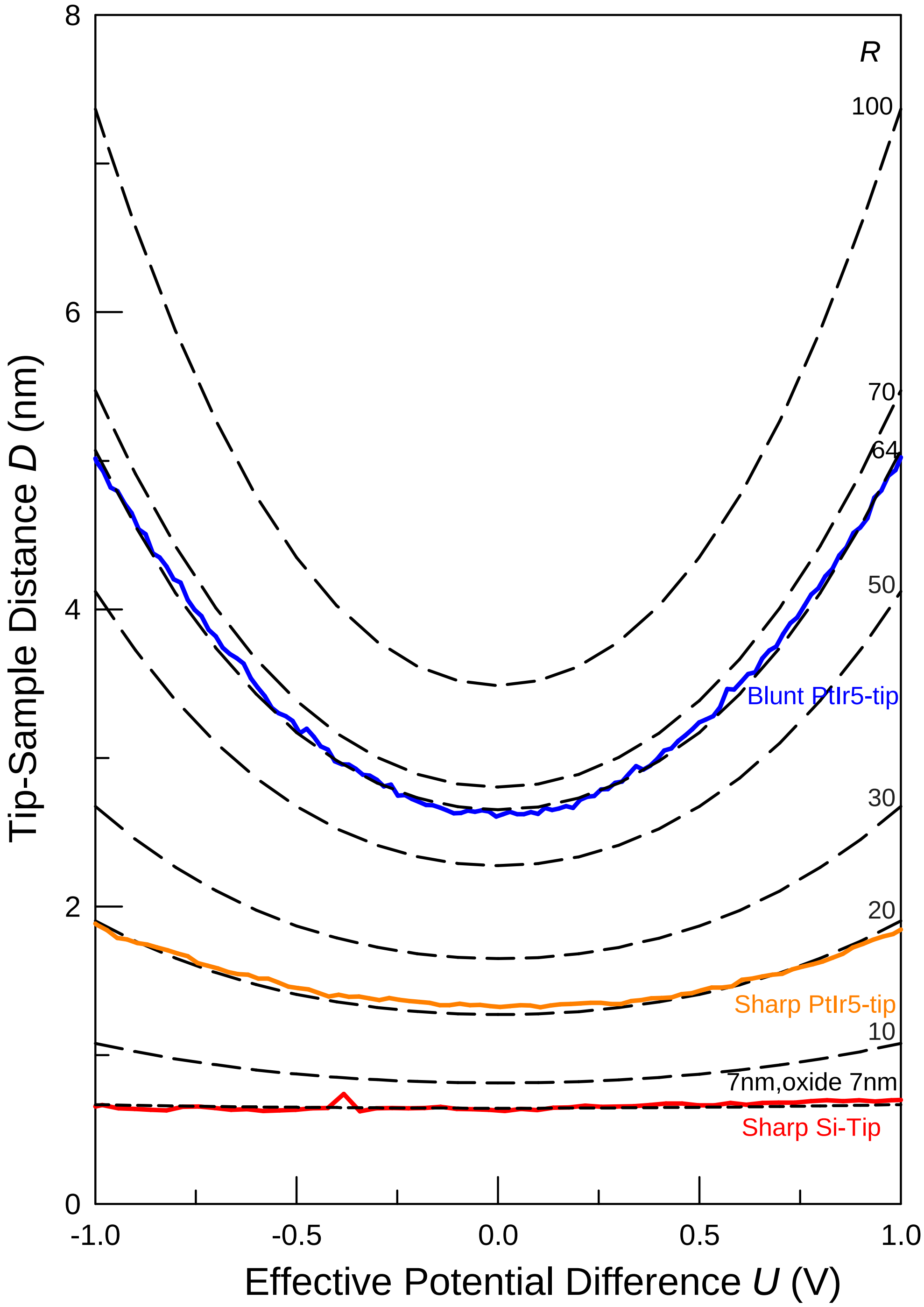


Fig. 5.11 Tip sample distance as a function of the effective potential difference. The different curves were obtained by different cantilevers have fitting with calculation from $D(V_t)$. For a sharp tip, the variation of D is small. For a blunt tip, the variation of D is large. The Radius of sharp Si-tip, sharp PtIr5-tip, and blunt PtIr5-tip are 7, 20, and 64 nm, respectively.

5.3.3 The step height versus effective potential difference

(a) Sharp PtIr5-tip

The void height versus tip bias can be plotted from the calculation of the $D(V_t)$. The CPD is 0.5 V which calculated from $D(V_t)$. The fitting for void height versus effective potential difference of a sharp PtIr5-tip is shown in Fig. 5.12. The calculated plot is shifted upward 0.7 nm, and the value is the true SiO_2 thickness.

The calculated plot has a good fitting with experimental data between -1 V and 1 V. Exceeding ±1 V, the void height is lower than the calculation plot. It is because the void size is not large enough. The tip can be measured correct height for the far distance between tip and sample. For this sample, we only can discuss the bias range between -1 V and 1 V.

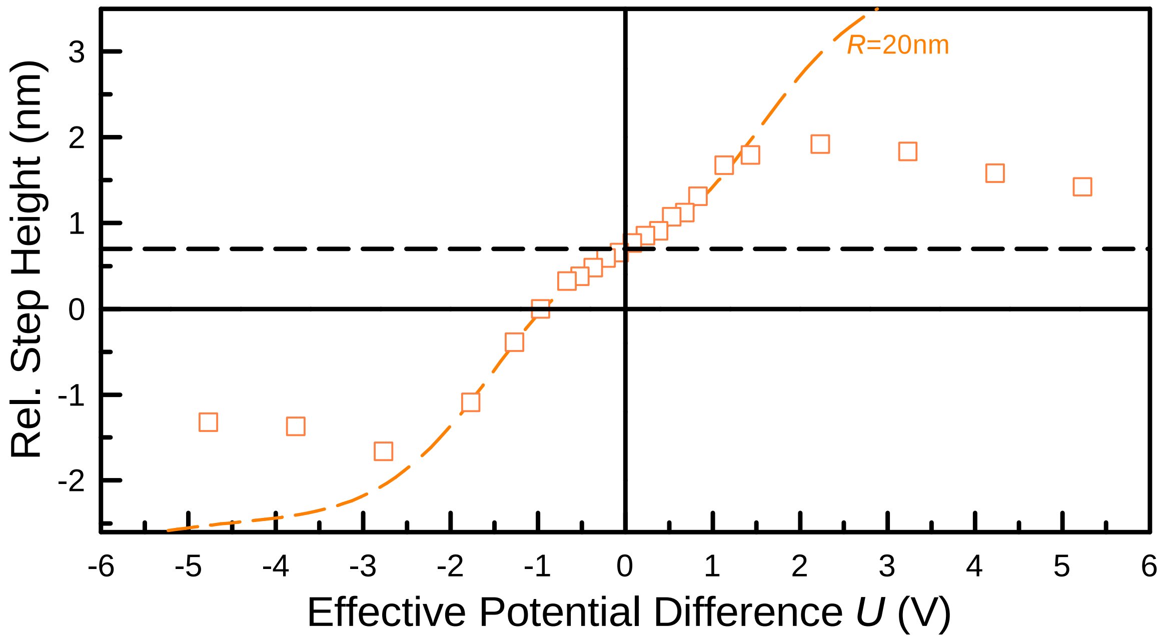


Fig. 5.12 The experimental data have good fitting with the calculation for R = 20 nm. There are bad fitting over 1 V, when electrostatic force is large enough.

(b) Relative step height versus effective potential difference

Figure 5.13 showed the three series of void heights versus effective potential difference of the different tips. The Blunt-tip has more dependence with applying bias than the sharp-tip. The calculation plots are fitted with the three tips, and have good fittings between -1 V and 1 V. The step height is 0.7 nm and show the dash line in Fig. 5.13.

For sharp Si-tip, there are 7 nm native SiO_2 layer cap on the tip and the step height variation is fitted with the calculation as shown in Fig. 5.13(a). For sharp PtIr5-tip, the fitting-radius is about 20 nm, there are good fitting in the low bias as shown in Fig 5.13(b). At the higher bias, the measuring heights are lower than the calculation. The deviations are cause by the small SiO desorption-size. For blunt PtIr5-tip, the fitting-radius is about 64 nm as shown in Fig.5.13(c). There are narrower ranges for step-height fitting than the sharp PtIr5-tip. The ranges of good calculation fitting are wide for sharp tip. It is because the sharp tip can reduce the effect of the electrostatic force, and the void heights are nearer the real void's height.

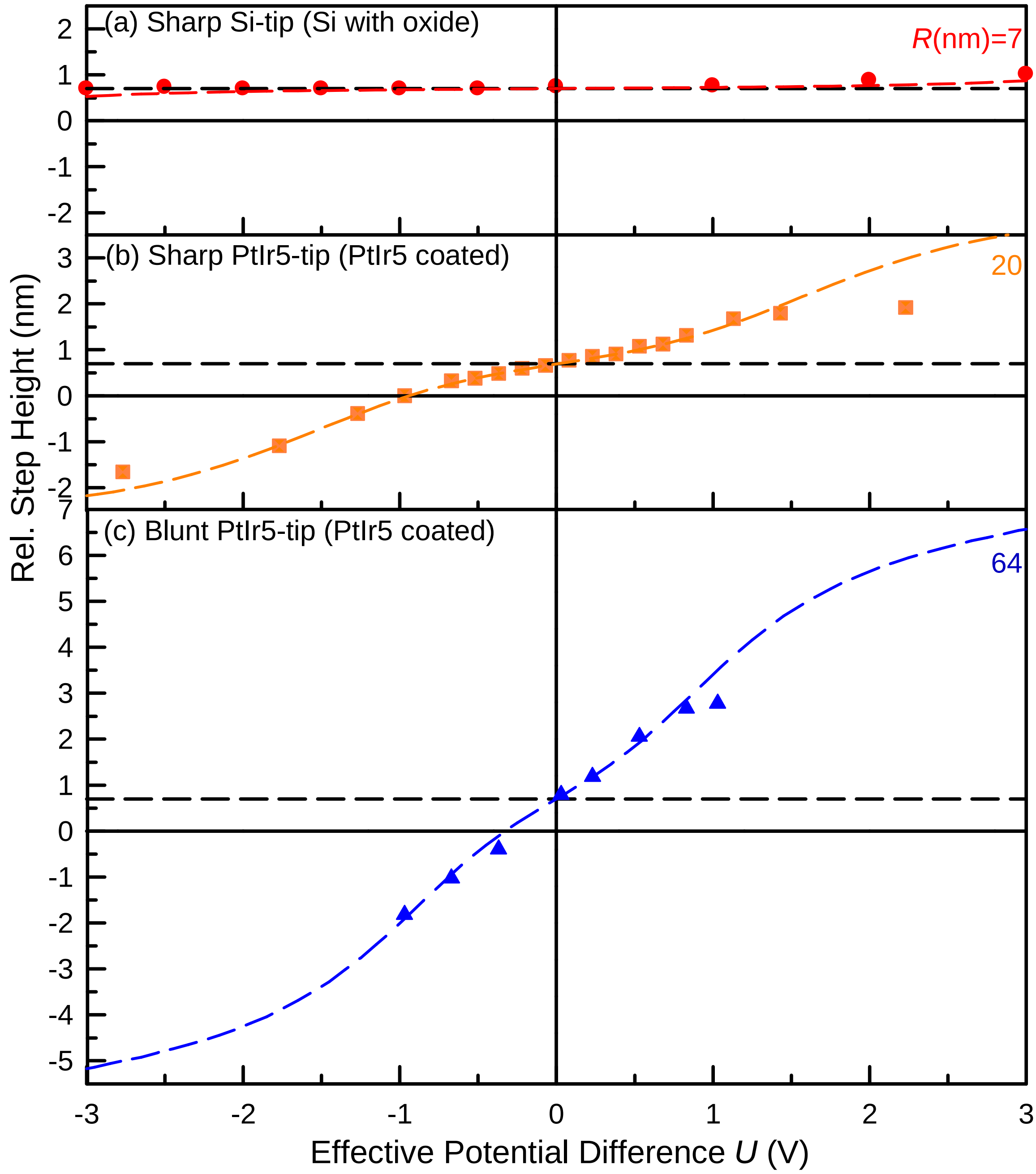


Fig. 5.13 The relative step heights obtained from the AFM images between the SiO_2 and Si(111) regions. The solid curves are fitted from Fig. 5.11 which has 0.5 V of the CPD. The tip radii used for the calculation are indicated. The calculation for the curve in (a) include the effect of an oxide layer of 7 nm covering the tip.

5.4 Conclusion

In this experiment, the ultra-thin SiO_2 layer can be performed by thermal oxide and measured by NC-AFM. The thickness of the ultrathin SiO_2 layer is about 0.7 nm. The CPD between SiO_2 layer and Si(111) is 0.5 V. The AFM images can be obtained by using different tip, and there are different variations of the void heights with different applying bias. The different void height is strong dependence with the different tip radius. The height variation of the Blunt-tip is larger than the sharp-tip with the applying bias.

The $D(V_t)$ curve is an another good method to obtain the tip radius instead of SEM micrograph. For sharp tip, the variation of the $D(V_t)$ curve is small, and for blunt tip the variation of the $D(V_t)$ curve is large. The real void's height can be obtained when tip bias is applied with the middle of the contact potential between Si(111) and SiO_2. For the void height versus tip bias, the experimental data and calculation from $D(V_t)$ for the different radius can usually be obtained a good fit between -1 V and 1 V.

Over ± 1 V, we need to consider the larger tip radius will cause larger electrostatic force. The distance between tip and sample is increasing with increasing bias. The AFM image is becoming vague gradually with increasing tip-sample bias. The large tip radius has more influence than the small tip radius. The void height is different to measure correctly during the high bias, besides the void size is only about 100×100 nm^2.