

# Oxygen Enhanced Surface Roughening of Si(111) Induced by Low-Energy Xe<sup>+</sup> Ion Sputtering

Takuya Miyagawa, Kousuke Inoue, and Masahiko Inoue\*  
Dept. Electric & Electronic Eng., Facult. Eng., Setsunan Univ.  
17-8 Ikedanakamachi, Neyagawa, Osaka 572-8508, Japan  
\*m-inoue@ele.setsunan.ac.jp

(Received: January 24, 2009; Accepted: February 16, 2009)

The secondary electron spectra from the Si (111) surface induced by irradiation of Xe<sup>+</sup> ions of 300 eV were measured as functions of oxygen exposure time. The intensity of ion-induced secondary electrons decreased with the increase of oxygen exposure time for Xe<sup>+</sup> irradiation, while the intensity of electron-induced secondary electrons increased. The atomic force microscopy (AFM) observation supported that the decrement of the peak intensity and the peak energy of the ion-induced secondary electron spectra are originated from the surface roughening enhanced by oxidation. This suggests that the ion-induced secondary electron spectroscopy can be applied for the real-time monitoring of the surface roughening during ion sputtering as a complementary method to AFM or scanning tunneling microscopy.

## 1. Introduction

For the high resolution sputter depth profiling, the low-energy ion beam is often used to reduce the atomic mixing caused by ion bombardment. It is, however, well known that the ripple topography is often produced on a Si surface by low-energy O<sub>2</sub><sup>+</sup> ion sputtering in the energy region of a few hundred eV, leading to the degradation of the depth resolution in secondary ion mass spectrometry analysis. This phenomenon depends on sputtering conditions such as ion species, incident energy, incident angle, oxygen concentration at the surface, but the mechanism of the ripple formation has not been fully clarified yet. As the secondary electron emission induced by ion bombardment is affected by surface topography as well as surface composition, there is a possibility that the dynamic roughening process of the surface may be monitored by the ion-induced secondary electron spectroscopy.

To confirm this, in the present study, we measured ion-induced secondary electron spectra from the Si(111) surface induced by irradiation of Xe<sup>+</sup> ions of 300 eV as a function of oxygen exposure time. The results revealed that the intensity of ion-induced secondary electrons decreased with the increase of the oxygen exposure time for Xe<sup>+</sup> sputtering, while the electron-induced secondary electron intensity increased. Atomic force microscope (AFM) observation supported that the decrement of the peak intensity and the peak energy of the secondary electron spectra are originated from the surface roughening enhanced by oxidation.

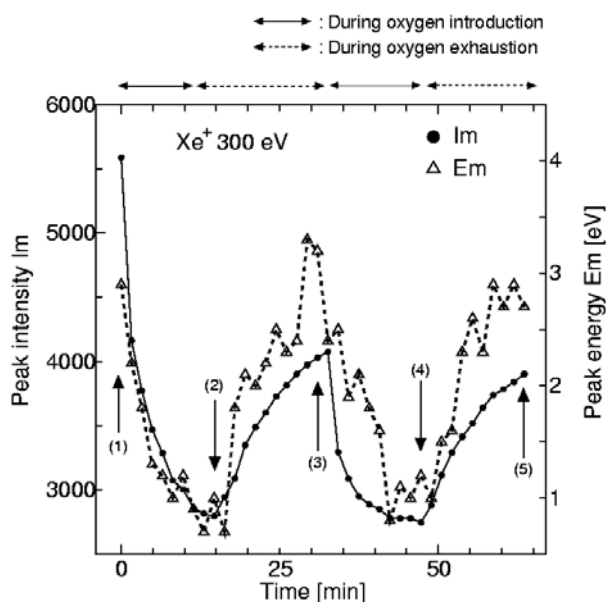
## 2. Experimental

Experiments were carried out using a scanning Auger electron microprobe (JAMP-10, JEOL) equipped with a floating-type low-energy ion gun [1,2]. The base pressure in the analysis chamber of the JAMP-10 was ~ 7 x 10<sup>-8</sup> Pa. The incident angle of the ion beam was 60 degrees from the surface normal. Focusing and alignment of the ion beam were carried out using a co-axial sample stage and a dual nano-ampere meter [3]. The ion energy was 300 eV. The ion current density was 26 nA/mm<sup>2</sup>, and the beam spot size was 2.4 mm (FWHM) estimated from the beam broadening parameter, BBP [4]. Si(111) samples were cleaned by HF(10%) etching to remove a native oxide layer before experiments. Secondary electron spectra from the Si(111) surface induced by Xe<sup>+</sup> ion bombardment were measured with a cylindrical mirror analyzer during two alternate cycles of oxygen introduction and exhaustion. The detection angle of the secondary electron was from 0 to 42.3 degrees from the surface. The pressure in the analysis chamber during oxygen introduction was ~ 1.5 x 10<sup>-5</sup> Pa.

## 3. Results and Discussion

Figure 1 shows the change in the peak intensity I<sub>m</sub> and the peak energy E<sub>m</sub> of the Xe<sup>+</sup> induced secondary electron spectra from Si(111) surface as a function of time of oxygen introduction and exhaustion. It was found that the ion-induced secondary electron intensity decreased with the increase of the surface oxygen concentration. Auger spectra in Fig.2 show that the sample surface was fully oxidized at the point (2) indicated in Fig.1,

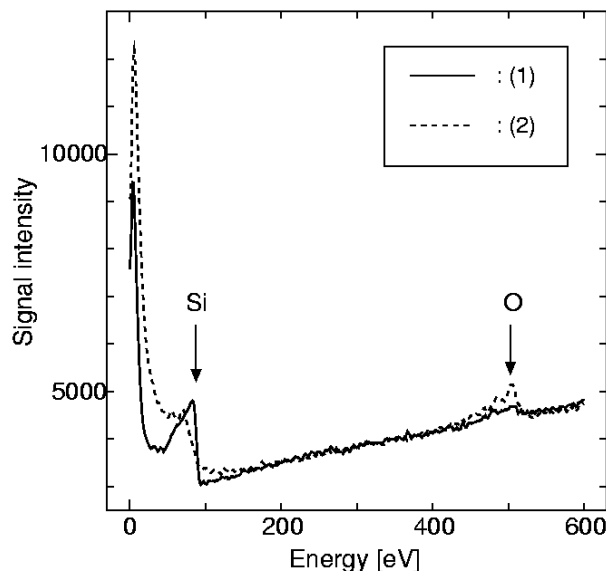
while the almost clean Si surface was revealed at the point (1). At points (3) and (4), almost the same spectra as (1) and (2) were obtained, respectively. Note that the intensity of electron-induced secondary electrons is enhanced by oxidation as confirmed in Fig.2. For the comparison, the secondary electron spectra induced by irradiation of 300 eV He<sup>+</sup> ions were measured in the same way as functions of oxygen exposure time. Results showed that the peak energy decreased with the increase of the surface oxygen concentration as same as the case of Xe<sup>+</sup> bombardment, but the peak intensity increased oppositely. Therefore, the reduction of the intensity of Xe<sup>+</sup> ion-induced secondary electrons in Fig.1 can hardly be considered to be only due to the chemical effect.



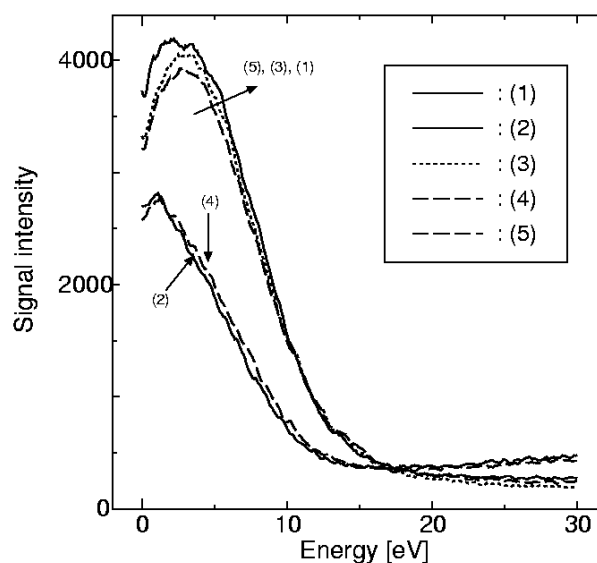
**Fig. 1** Change of the peak intensity  $I_m$  and the peak energy  $E_m$  of Xe<sup>+</sup> bombardment induced secondary electron spectra during oxygen introduction ( $\sim 1.5 \times 10^{-5}$  Pa) and exhaustion.

Figure 3 shows the Xe<sup>+</sup> bombardment-induced secondary electron spectra obtained at the points from (1) to (5) indicated in Fig.1, respectively. At the fully oxidized points of (2) and (4), the higher energy part of the secondary electron spectra obtained at the non-oxidized points of (1), (3) and (5) is reduced. This can be explained by the surface topographic effect. The ranges of 300 eV Xe<sup>+</sup> and He<sup>+</sup> in Si sample are estimated to be about 2.4 and 4.2 nm by SRIM Monte Carlo simulation [6], respectively. Projected ranges of these ions become half of these values for the incident angle of 60 degrees. The attenuation lengths of secondary

electrons around the peak energy evaluated by Seah and Dench's equation [7] are rather longer than these ranges as confirmed in Fig.4, in particular, in the low energy region less than around 20 eV.



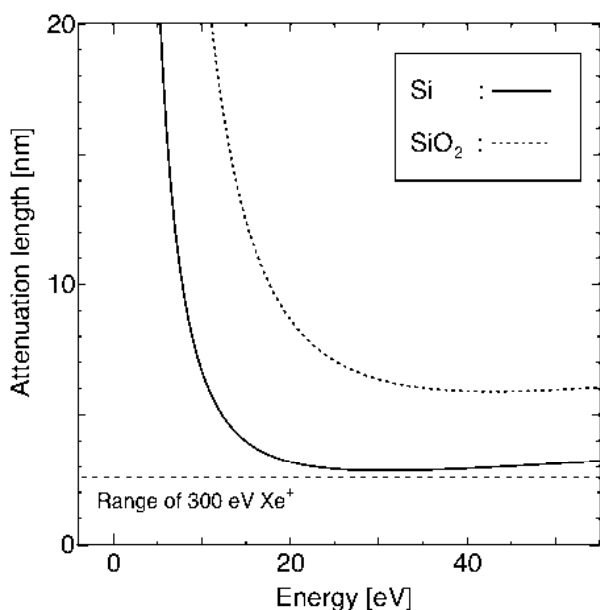
**Fig. 2** Auger electron spectra obtained at the points (1) and (2) indicated in Fig.1, respectively.



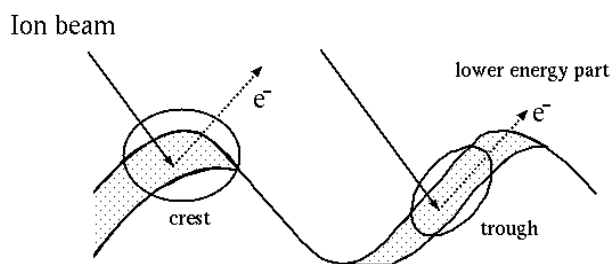
**Fig. 3** Xe<sup>+</sup> bombardment-induced secondary electron spectra obtained at the points from (1) to (5) indicated in Fig.1, respectively.

Figure 5 shows the schematic drawing for the ion-induced secondary electron emission from the roughened surface. In the case of a flat surface, secondary electrons are emitted from the whole surface. But in the case of a roughened surface with the larger amplitude than the range of the incident

ion, most of secondary electrons are emitted from crests of the ripple. Therefore, the secondary electron intensity decreases. Furthermore, secondary electrons emitted from troughs travel relatively longer distance, and, therefore, contribute to a lower energy part of the spectra. These lead to the decrement of the peak intensity and the peak energy as shown in Fig.3.



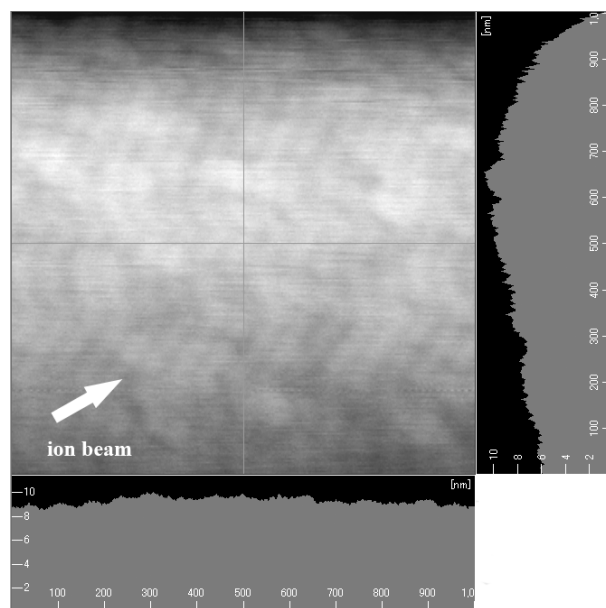
**Fig. 4** Attenuation lengths of secondary electrons evaluated by Seah & Dench's equation [7] in Si and SiO<sub>2</sub> samples.



**Fig. 5** Schematic drawing for the ion-induced secondary electron emission from roughened surface with the larger amplitude than the escape depth of the secondary electron.

In order to investigate the adequacy of this model, the AFM (VN-8000, KEYENCE) observation was carried out. Figure 6 shows the AFM image of the Si(111) surface obtained after sputtering by 300 eV Xe<sup>+</sup> of 85 nA/mm<sup>2</sup> for 4 hours without oxygen introduction, corresponding to the condition at the point (1) indicated in Fig.1. The scanned area was 1000x1000 nm<sup>2</sup>. The height profiles h(x) and h(y)

along the horizontal and vertical lines marked in the image are shown at the bottom and right, respectively. The relatively flat surface with the roughness amplitude of less than a few nm was observed. On the other hand, Fig.7 shows the AFM image of the Si(111) surface obtained after sputtering by 300 eV Xe<sup>+</sup> of 85 nA/mm<sup>2</sup> with oxygen introduction (~1.5 x 10<sup>-5</sup> Pa) for 4 hours. Although the periodic ripple structure could not be observed, the remarkable surface roughening with the amplitude of about 5 to 10 nm, and the wave length of ~ 100 nm was clearly observed.

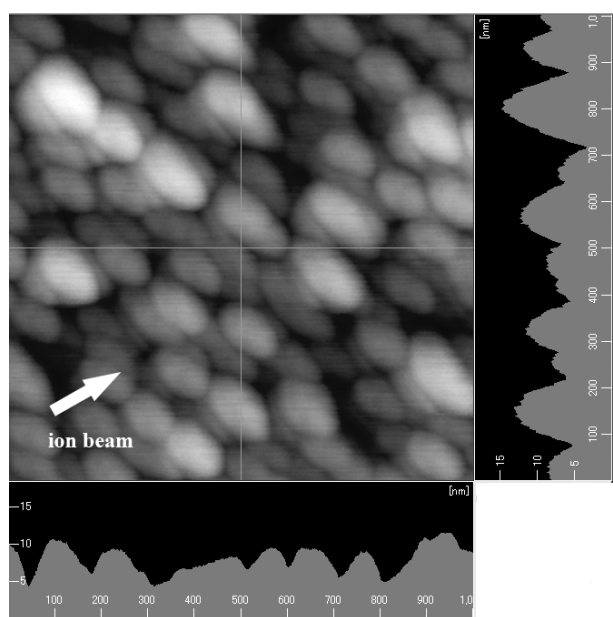


**Fig. 6** AFM image of the Si(111) surface obtained after sputtering by 300 eV Xe<sup>+</sup> 85 nA/mm<sup>2</sup> without oxygen introduction for 4 hours, corresponding to the condition at the point (1) indicated in Fig.1. The scanned area is 1000x1000 nm<sup>2</sup>. The height profiles h(x) and h(y) along the horizontal and vertical lines marked in the image are shown at the bottom and right, respectively.

According to Bradley and Harper's theory of the ripple structure formation induced by ion bombardment [5], periodic perturbations with the smallest wavelengths grow most rapidly due to the curvature dependence of the sputtering yield, but are reduced most rapidly due to surface relaxation effects such as the surface self-diffusion, leading to the ripple structure with the specific wavelength determined by the balance of sputtering and surface self-diffusion.

In the case of sputtering during oxygen introduction, the surface self-diffusivity is rather

reduced by surface oxidation, and, therefore, the stable wavelength seems to be also reduced to ~100 nm. These AFM observation results support the model of Fig.5. This suggests that the ion-induced secondary electron spectroscopy can be applied for the real-time monitoring of the surface roughening process during ion sputtering as a complementary method to AFM or scanning tunneling microscopy.



**Fig. 7** AFM image of the Si(111) surface obtained after sputtering by 300 eV Xe<sup>+</sup> of 85 nA/mm<sup>2</sup> with oxygen introduction ( $\sim 1.5 \times 10^{-5}$  Pa) for 4 hours, corresponding to the condition at the point (2) indicated in Fig.1. The scanned area is 1000x1000 nm<sup>2</sup>. The height profiles  $h(x)$  and  $h(y)$  along the horizontal and vertical lines marked in the image are shown at the bottom and right, respectively.

#### 4. Summary

The secondary electron spectra from the Si (111) surface induced by Xe<sup>+</sup> ions of 300 eV were measured as functions of time during two alternate cycles of oxygen introduction and exhaustion. Both

of the peak intensity and the peak energy of ion-induced secondary electron spectra from the fully oxidized Si(111) surface are reduced due to the reduction of the higher energy part of the spectra observed for that from the clean Si surface. This decrement can not be explained only by chemical effects because the peak intensity of the electron- and 300 eV He<sup>+</sup> ion-induced secondary electron spectra are both enhanced by the surface oxidation. AFM observation revealed that the surface roughness increased to ~ 100 nm by surface oxidation; this is probably due to the reduction of the surface self-diffusivity of Si. The reduction of the higher energy part of ion-induced secondary electron spectra can be explained by the surface topographic effects. This suggests that the ion-induced secondary electron spectroscopy can be applied for the real-time monitor of the surface roughening process during ion sputtering as a complementary method to AFM or STM.

#### 5. Acknowledgements

The authors thank Dr. Y. Kimura of Osaka University and Dr. Y. Matsuo of Setsunan University for their help on AFM observation.

#### 6. References

- [1] Y.Mizuhara, J.Kato, T.Nagatomi, Y.Takai, T.Aoyama, A.Yoshimoto, M.Inoue, R.Shimizu, Surf. Interface Anal. **35**,382(2003).
- [2] R.Shimizu, and M.Inoue, J. Surf. Anal. **10**,154(2003).
- [3] M.Inoue, K.Kurahashi, and K.Kodama, J. Surf. Anal. **10**,197(2003).
- [4]<http://www.ss.teen.setsunan.ac.jp/bbp2004/e-bb-pjava.html>
- [5] R.M. Bradley and J.M.E. Harper, J. Vac. Sci. Technol. **A6**, 2390(1988).
- [6] James Ziegler – SRIM & TRIM, <http://www.srim.org/>
- [7] M.P. Seah and W.A. Dench, Surf. Interface Anal. **1**, 2(1979).