Energy-Filtered Image of Surface Blisters by Grazing Incidence Electron Microscopy

Sin Igarashi¹, Shunsuke Muto¹, Tetsuo Tanabe¹ and Tadashi Maruyama²

¹ Center for Integrated Research in Science and Engineering, Nagoya University, Nagoya 464-8603, Japan
² The Wakasa Wan Energy Research Center, Tsuruga 914-0192, Japan

We have examined fine structure of surface blisters formed by D⁺ or He⁺ irradiation onto mono-crystalline silicon by grazing incidence electron microscopy (GIEM), using an energy-filtering transmission electron microscope (EFTEM). Mapping of the projected thickness clearly visualized the structural difference of the blister skins formed by D⁺ and He⁺ irradiation respectively. A He distribution image of the He-blisters was also successfully obtained.

(Received June 8, 2001; Accepted August 10, 2001)

Keywords: silicon, ion irradiation, energy filtering transmission electron microscopy, elemental distribution image

1. Introduction

Recent development of electron energy-loss spectroscopy (EELS) in cooperation with transmission electron microscopy (TEM) has extended its capability to explore not only local microstructures but also electronic structures of the specific submicron areas. In this respect, we have developed grazing incidence electron microscopy (GIEM),¹ based on reflection electron microscopy (REM) and EELS, which enables us non-destructive analysis of surface protrusions of non-ductile materials. We have applied this method to study surface blistering of mono-crystalline Si produced by gas ion irradiation.² The GIEM and associated electron diffraction and EELS successfully revealed the shape, size and density of the blisters, as well as construction of the blister skins.

In the present letter we report the fine structure of the blister skins as two-dimensional projected images by applying energy-filtering TEM (EFTEM) to GIEM.

2. Experimental

The experimental setup of GIEM has been described in detail previously.¹–³ The sample was a 1 mm x 1 mm x 2 mm rectangular block of Si crystal. Each block was mounted on a molybdenum single hole mesh with shiny (100) face edge-on. The shiny polished face of the sample was irradiated at room temperature by D⁺ (incident ion energy E₀ = 10 keV) or He⁺ (E₀ = 16 keV) to give the nearly same projected range (~200 nm). The dose φ was 1.0 x 10²² m⁻² for D⁺ and 2.0 x 10²² m⁻² for He⁺ irradiation to obtain a fully-grown blister size for easy observation. Observation was performed with JEOL JEM-3000F equipped with a post-column type Gatan Imaging Filter (GIF) using a charge coupled device (CCD) detector with 1024 x 1024 pixels.

3. Thickness Mapping

The specimen thickness t, through which the incident electrons travel, is given by

\[ t/\lambda = \ln(I/I₀) \]  

where I₀ is the zero-loss (elastic scattering) intensity, I is the total electron intensity reaching the spectrometer and λ is the electron mean free path for energy-loss.⁴ If taking two images with only elastically scattered (zero-loss image) and the totally scattered electrons (unfiltered image) respectively, and then computing the natural logarithm of the intensity ratio of the two images on each image pixel in the same way as eq. (1), each resultant value is proportional to the projected sample thickness at this pixel position.

In the present case, the electron path length must be counted through the blister skins and internal gas molecules. Considering the internal gas pressure of the blisters,² the atomic density of gas molecules could be estimated to be one order of magnitude less than the solid blister skins. Furthermore, the main energy-loss process in the internal gas region, which contributes the energy-loss spectra, is ionization of 1s electrons, whose cross section is very small for the present high-energy incident electrons.⁵ It is therefore considered that the electron mean free path in the internal gas region is at least one order of magnitude greater than that of solid silicon. In other words, the derived thickness from eq. (1) can be taken as the thickness of the blister skins only within the present experimental accuracy.

The intensity distributions actually correspond to the expected projected thickness of the blister skins and the internal gas molecules have little influence on them, which supports the above-mentioned interpretation. The thickness mapping of D-blister and He-blister thus obtained is shown in Fig. 1. It is clearly seen that the D-blister skin has comparatively uniform thickness, whereas the He-blister skin has sponge-like structure containing a high density of pores. This corresponds to the fact that the He-blister was formed simply by the accumulation of implanted gas and the subsequent coalescence of gas bubbles, whereas in the D-blister the formation of Si-D...
bonding was triggered in the large crack formation along the projected range.\textsuperscript{2) The detailed blistering mechanism will be discussed elsewhere.

4. He Distribution Image

An attempt was made to image the He gas distribution using the He K-absorption edge (24.6 eV\textsuperscript{4}). As seen in the spectrum from the He-blister (Fig. 2 in Ref. 2), the He K-edge is superposed on the slightly higher energy side of the large first plasmon peak of Si (16.7 eV\textsuperscript{4}). This makes He-imaging by the conventional three-window method difficult because the pre-edge profile cannot be modeled by the power-law.

We hence resorted to the two-window method, or jump-ratio method.\textsuperscript{5) He distribution image is shown in Fig. 2, which is obtained with the energy-selecting slit of 2 eV in width with an enhanced contrast. The unrealistic contrast just outside the blister periphery was likely caused by the deviation of detector crossover point due to the very narrow slit width. Nevertheless, the image intensity is highest around the center of the blister, reflecting the longest electron path length in the He-filled internal dome. We therefore conclude that the image correctly reflects the projected He-gas distribution. Note that the He-image appears bright even at the blister skin area. This indicates that the He-blister skin contains a considerable amount of He presumably in the form of small bubbles.

5. Summary

Energy-filtered imaging of internal fine structures of blisters on D\textsuperscript{+}- or He\textsuperscript{2+}-irradiated Si surface was successfully conducted. Mapping of the projected sample thickness indicated that the D-blister skin had rather uniform thickness, whereas the He-blister skin showed sponge-like structure, reflecting the different formation mechanism for each type of blisters. Furthermore, the He distribution image showed that the blister skin contains a considerable amount of He in the form of gas bubbles.

REFERENCES