Luminescence from SiO₂ by Helium Ion Microscopy without any Damage Characterized by TEM-EELS

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Abstract

Luminescence from a SiO_2 sample was investigated using helium ion microscope, and generation of the luminescence was confirmed. TEM-EELS showed no damage in the sample after the helium ions beam irradiation for the luminescence characterization.

1. Introduction

We have reported a secondary electron (SE) mode imaging with less damage and less transformation of soft materials such as low dielectric materials and photo resist fine patterns for semiconductor processing by the helium ion microscopy (HIM) [1], and explored possibility of helium ion beam induced luminescence for the first time [2]. However it was not clear whether it really was luminescence or not, and the damages during the helium ions irradiation in the samples for the characterization have not been studied.

In this study, after helium ions beam irradiation onto a SiO_2 film and Si substrate using the HIM under standard helium ions beam irradiation conditions for the luminescence characterization, damages in the samples were characterized by a transmission electron microscopy (TEM) electron energy loss spectroscopy (EELS) method [3].

2. Experiments

Helium ion beams of a few pA, in which a dose amount was $1E13/cm^2 - 5E14/cm^2$, which are typical HIM SE mode observation conditions, was irradiated onto the SiO₂ film of 400 nm thick and Si substrate at 30 kV, and generation of luminescence at wavelength of 300 - 800 nm was studied. And then those SiO₂ films and Si substrates were examined by the TEM-EELS method. As a reference, the SiO₂ film was examined by a cathode luminescence (CL) using a secondary electron microscope (SEM) at around 1 nA, 5 – 15 kV.

3. Results and Discussions

In the SEM-CL case, two peaks at 458 and around 655 nm were observed as shown in Fig. 1(a), while in the HIM luminescence case, three peaks at 281 nm, 447 nm, and 672 nm were observed as shown in Fig. 1(b). The CL peaks at lower wavelength might be missing because of the higher intensity peak at 655 nm in the case of 5 kV. It has been

reported that stress shows a shift in the CL spectra [4], [5] and further discussion is required for the difference in the wavelength between those two results by the SEM and the HIM. As shown in Fig. 1(b), in the HIM luminescence case, the shift in the spectra from the SiO_2 with different doses was not seen, while the intensity became higher with the higher dose. This probably means no damage was generated during the helium ions irradiations. The peak of 447 nm is quite similar to an atomic spectrum from exited helium atoms, which are possibly generated at the helium ions beam collision onto the sample, while with a higher spectrum resolution measurement, width of the broad peak of 447 nm did not narrow, so it presumably be the "luminescence", not the atomic spectrum.

Energy loss near edge structure (ELNES) and valence EELS (V-EELS) spectra at distances of 20, 100, 250, and 400 nm from SiO_2 surfaces were shown in Fig. 2(a), (b). ELNES and V-EELS give us information of electronic structure of conduction band, and defect states in the band gap, respectively. A project range of irradiated helium ions was 250 nm and it might be damaged the heaviest, if such damage was generated. Any difference in the spectra was not observed among all spectra of the ELNES and V-EELS. Similar experiments were performed for a Si substrate, and quite the same results have been obtained without any detectable damage or bubble in the sample. It has been revealed that no damage generated during the HIM observations and luminescence characterization at standard helium ion beam irradiation conditions within a detection limit of the TEM-EELS.

4. Summary

In the HIM characterization technology, as a new application, "luminescence method" was shown and it was confirmed that the helium ion beams did not generate any detectable damage in the SiO_2 sample.

Collisions between helium ion beams and materials, SiO_2 in this study, including damage generation in the materials have not been well understood, and further discussions, especially on the mechanism of the helium ion beam luminescence generation, will be required for further applications.

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References

[1] S. Ogawa, et al, Jpn. J. Appl. Phys., 49 04DB12 (2010)

[2] S. Ogawa, et al, Proc. of International Interconnect Technology Conference P1.31 (2011)

[3] Y. Otsuka, et al, Jpn. J. Appl. Phys., 49 111501 (2010)

[4] Mc Clure, et al, pp. 1-132 in Treatise on Solid State Chemistry, Vol. 2. Edited by N. B. Hannay. Plenum Press, New York, 1988

[5] M. Kodera, et al. Proc. of International Interconnect Technology Conference, p.212 (2005)





Fig.1 Spectra from SiO_2 by the SEM CL at 5, 15kV (a) and the helium ion beam induced luminescence at different ion doses of 1.3-20 E13/cm² at 30kV (b)

Fig.2 TEM ELNES and V-EELS spectra at distances of 20, 100, 250, and 400 nm from the SiO_2 surface without helium ions beam irradiation (a) and $2E14/cm^2$ helium ions beam irradiation (b).