Thin Films and Coatings

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TEM/STEM EELS investigation of silicon oxidation under composite ion beam irradiation at room temperature

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Recent time we have developed new technique to oxidize silicon by means of composite ion beam irradiation at room temperature using composite (protons and oxygen) ions ion beams with oxygen concentration ~10^{-4} [1]. To understand the nature of irradiation-induced oxidation at room temperature in this work we performed cross-section EELS TEM/STEM study of microstructure evolution for different ion fluences and irradiation conditions.

The mono-crystal Si (100) wafers were irradiated at room temperature by ions which were extracted from RF plasma by 1 keV pulse HV bias. Composition of the ion beam was controlled by the partial pressure of different gases in discharge chamber. The cross section samples were prepared by the conventional technique by Ar ion milling. The degree of silicon atoms oxidation state was controlled by the position of Plasmon peak of energy loss. EELS spectra were taken in STEM mode to get local information with small probe size.

At the first case we made a gas mixture from pure hydrogen and oxygen gases after deep camber aging ("dry" oxidation). The ion beam composition was controlled by the partial pressure of dry oxygen gas. At this case we have found the formation of porous silicon at the fluence of ~10^{18}cm^{-2} (Figure 1). According to EELS data analysis at this stage of irradiation the thickness of oxide layer did not exceed a few nanometers from the surface. Thus the nano-porous silicon was formed at these irradiation conditions. With increasing of the fluence the oxide thickness increased that is proved by the EELS study across the surface of the sample, but the average pores diameter dropped down and at ~ $1.1 \cdot 10^{20}$cm^{-2} pores disappeared at all and perfect silicon oxide layer was formed.

The second way to form the composite ion beam was to use residual gases in the discharge chamber while the rest was filled with hydrogen ("wet" oxidation). In this case the main residual gas was water because it was really difficult to get water-free conditions without special aging procedures. We didn't find big porous formation in silicon during irradiation in this case. The oxidation was quicker, probably due to the higher diffusion rate of OH ions because their small size, but the final thickness of SiO_2 (in both "dry" and "wet" cases) corresponded to the hydrogen projected range (Figure 2).

To study the depth dependence of pores formation region we performed samples irradiation with 3.8 keV energy ions at "dry" oxidation regime. The depth of pores formation for this energy of ions is more than the corresponded depth for 1keV irradiation (Figure 3). EFTEM O jump ratio map showed non uniform depth distribution of oxidation (Figure 4). The maximum oxygen concentration was observed up to 20 nm from the surface. In this region the silicon was fully oxidised (Si^{+4}) what was proved by the Plasmon peak position at EELS spectra (Figure 5). With increasing of the depth the Plasmon peak position smoothly moved to 16 eV (Figure 5) which corresponds to the virgin silicon (Si^0).

According to our understanding the proton induced vacancy production process played an important role during irradiation-induced silicon oxidation under composite ion beam irradiation. We can compare the experimental integrated EFTEM jump ratio oxygen distribution with the calculated one [2] (Figure 6). It is clear from Figure 6 that together with the presence of direct oxygen implanted area there is an inner maxima at the depth of preferable vacancy production from the proton component of ion beam. Due to the fact that we always observe the silicon oxidation to the depth up to the projected range of protons at high fluencies, we can conclude the radiation nature of the process of silicon oxidation at room temperature under composite ion beam irradiation.

Irradiation-induced silicon oxidation at room temperature can be used in wide range of nanotechnology applications (formation of sensors, transistors, etc.). We are grateful for financial support [3].

1. B.A.Gurovich, K.E.Prikhodko, E.A.Kuleshova, and D.A.Komarov, NIMB (2013). In Press (http://dx.doi.org/10.1016/j.nimb.2012.12.110)
2. Ziegler J.F., Biersack J.P., Ziegler M.D. The Stopping and Range of Ions in Matter, (Morrisville: Lulu Press Co., 2008) (http://www.srim.org)
3. This work is supported by the Russian Ministry of Science and Education (contract No.14.513.11.0008).

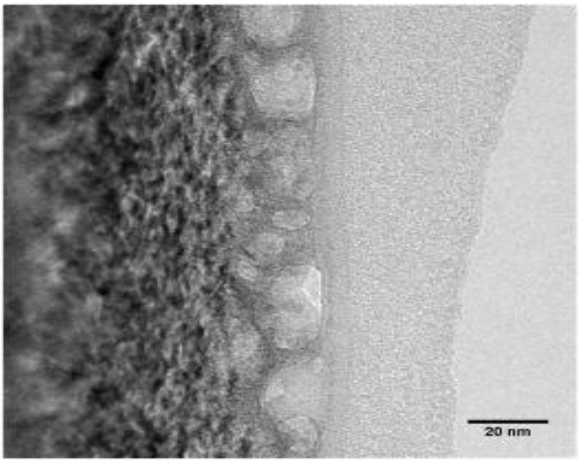

Figure 1. XTEM BF image of Si after 1 keV composite ion beam irradiation up to $9{,}4 \cdot 10^{17}$ cm^{-2} (“dry” regime).

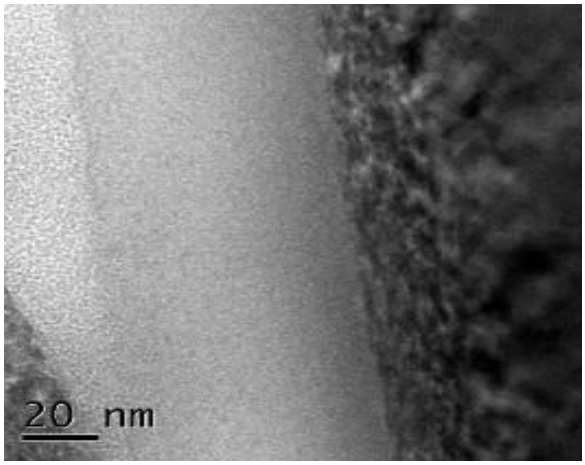

Figure 2. XTEM BF image of Si after 1 keV composite ion beam irradiation up to $1{,}2 \cdot 10^{20}$ cm^{-2} (“wet” regime).

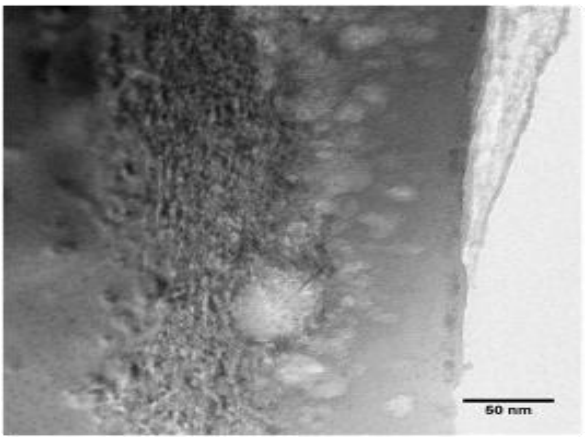

Figure 3. XTEM BF image of Si after 3.8 keV composite ion beam irradiation up to $2{,}9 \cdot 10^{19}$ cm^{-2} (“dry” regime).

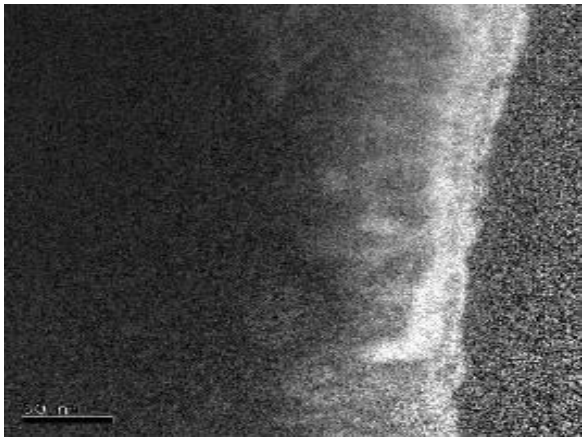

Figure 4. EFTEM oxygen jump ratio map of Si after 3.8 keV composite ion beam irradiation up to $2{,}9 \cdot 10^{19}$ cm^{-2} (“dry” regime).

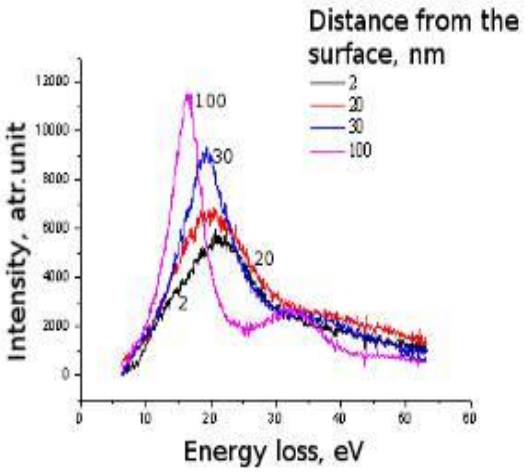

Figure 5. EELS Plasmon peak at different distances from the sample surface after 3.8 keV irradiation up to $2{,}9 \cdot 10^{19} cm^{-2}$ (“dry” regime).

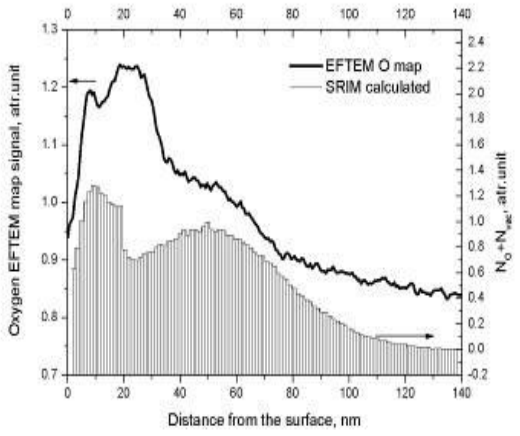

Figure 6. Integral EFTEM O signal from Figure 4 and calculated SRIM [2] distribution of normalised implanted O plus normalised vacancy after 3.8 keV composite ion beam irradiation (not to scale)