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Microstructure evolution during silicon oxidation at room temperature under composite ion beam irradiation

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ABSTRACT

In this work, we studied the silicon microstructure evolution during its oxidation under composite beam ion irradiation at room temperature. It was found that when the composite ion beam was formed by hydrogen and dry oxygen mixture at low doses ($\sim 10^{18}$ cm^{-2}), a porous silicon layer was formed. During irradiation, the pore size gradually reduced and at a dose of $\sim 10^{20}$ cm^{-2} pores disappear completely, and an uniform layer of silicon oxide was formed. If residual gases and hydrogen are used to generate a composite ion beam, the formation of porous silicon is not found. The final thickness of irradiation-induced silicon oxide corresponded to the projected range of protons at a given energy in both cases.

1. Introduction

We have recently developed a new technique to oxidize silicon by means of composite ion beam irradiation at room temperature using composite ion beams formed by protons and oxygen or OH ions [1]. This is the particular case of the selective attachment of atoms (SAA) process, which allows us to attach some atoms to the target by introducing them together with protons during composite ion beam irradiation. This target modification can be made to the thicknesses up to the projected range of hydrogen ions [2]. The typical used composition of the composite beams was (10^{-3}–10^{-4}). It was experimentally found that this composition region is optimal as a result balance between oxidation and sputtering processes competition [2].

Because of the different diffusion rate for O and OH ions we investigated two different processes of silicon oxidation under irradiation: "dry" (protons and O ions) and "wet" (protons and OH ions).

The main task of this work was to understand the nature of irradiation-induced silicon oxidation at room temperature under composite ion beam irradiation by means of cross-sectional electron energy loss (EELS) transmission electron microscopy/scanning transmission electron microscopy (TEM/STEM) study of microstructure evolution for these different ion beam compositions and doses.

2. Experimental

Mono-crystal n-type Si (100) wafers were irradiated at room temperature with ions extracted from RF plasma by pulses of (1–4) keV high voltage (HV) bias. The pulses were designed to eliminate the charging effect by applying a corresponding positive impulse at every period. This feature allowed us to perform irradiation of any insulators without charge effect. This was important because silicon oxidation during irradiation leads to a significant decrease of the target conductivity.

Composition of the ion beam was estimated by the partial pressure of different gases in discharge chamber [1,2].

Composite ion beam contained protons and oxygen ions and its composition (C) was controlled by the partial pressure of the oxygen or residual atmosphere at the discharge chamber during irradiation:

$$C_{O_2,H_2O} = \frac{P_{O_2,H_2O}}{P_{H_2} + P_{O_2,H_2O}} \quad (1)$$

In the case of "dry" process the ion beam composition (C_{O_2}) was estimated by the pressure of dry oxygen gas that was added to the chamber after long aging procedure. For "wet" process the pressure of residual gases (manly H_2O) was used to estimate the beam composition (C_{H_2O}) and the aging procedure of the chamber was not

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employed. Table 1 describes all irradiation conditions of the samples.

Samples for XTEM were prepared by the conventional technique of Ar ion milling. The oxidation degree of silicon atoms was controlled by the position of the plasmon peak in EELS using a post column spectrometer. EELS spectra were taken in STEM mode to get local information with small probe size. We also used the conventional energy-filtering TEM imaging technique (EFTEM) to visualize the depth distribution of oxygen atoms in the target.

3. Results and discussion

In the first case of "dry" oxidation the formation of porous silicon was observed at the fluence of $\sim 10^{18}$ cm^{-2} (Fig. 1a). The total

Table 1
Samples irradiation conditions.

Sample no.	Type of oxidation	Discharge chamber gas content	Beam composition	Ion energy, keV	Fluence, cm^{-2}
1	Dry	$H_2 + O_2$	4.9×10^{-4}	1	1×10^{18}
2	Dry	$H_2 + O_2$	4.9×10^{-4}	1	1×10^{19}
3	Dry	$H_2 + O_2$	4.9×10^{-4}	1	2.9×10^{19}
4	Dry	$H_2 + O_2$	4.9×10^{-4}	1	1.2×10^{20}
5	Wet	$H_2 + H_2O$	5.2×10^{-4}	1	1.2×10^{20}
6	Dry	$H_2 + O_2$	6×10^{-3}	3.8	2.9×10^{19}

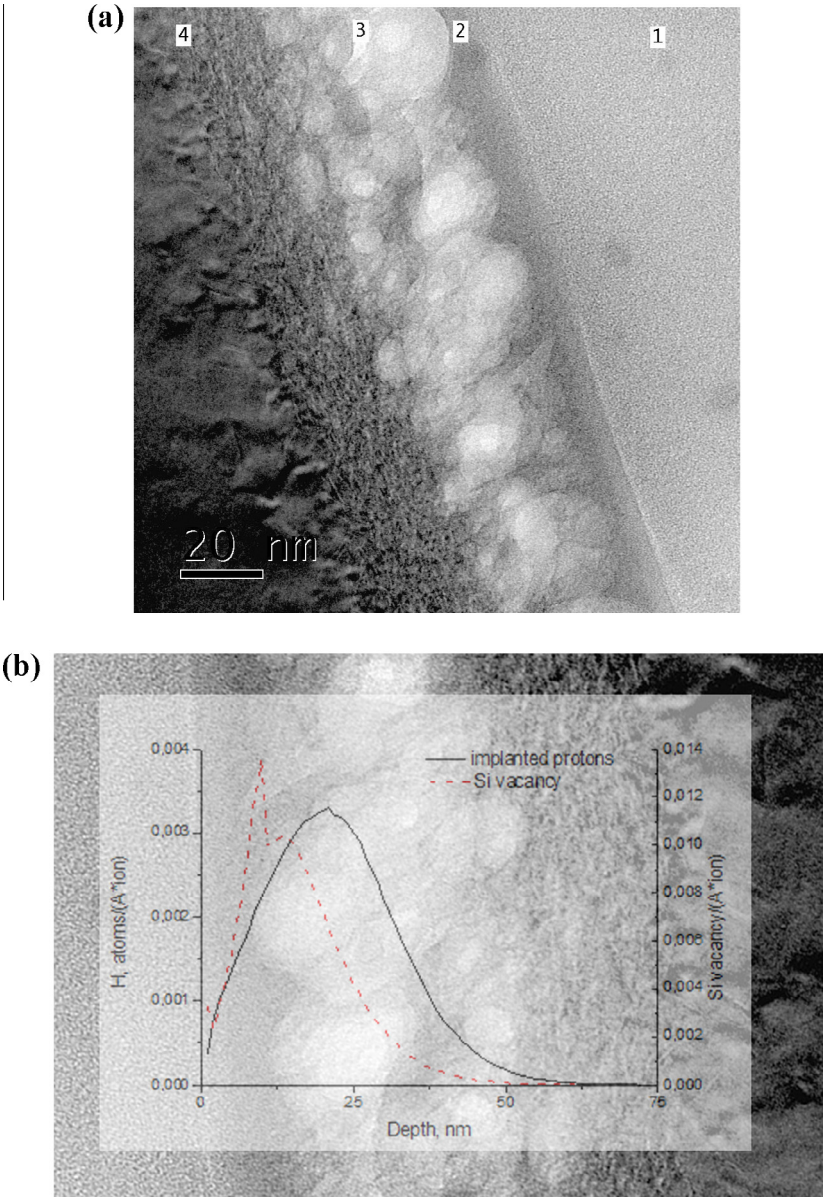

Fig. 1. (a) XTEM BF sample #1 image: 1 – glue, 2 – amorphous silicon, 3 – porous silicon, 4 – damaged substrate; and (b) – calculated vacancy and hydrogen ions depth distributions together with XTEM BF image of sample #1.