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# Irradiation effects on solid surfaces by water cluster ion beams

# Hiromichi Ryuto<sup>\*,1</sup>, Keiji Tada, Gikan H. Takaoka

Photonics and Electronics Science and Engineering Center, Kyoto University, Nishikyo, Kyoto 615-8510, Japan

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### ABSTRACT

The interaction between a water cluster ion beam and the surface of a silicon substrate was investigated. The sputtering yield of silicon by a water cluster ion beam was approximately ten times larger than that by an argon monomer ion beam. X-ray photoelectron spectroscopy was used to analyze the silicon surface irradiated with a water cluster ion beam. The analysis revealed that the surface was oxidized, and the oxidation was saturated approximately at the dose of  $1 \times 10^{14}$  ions/cm<sup>2</sup>. The number of disordered atoms measured by the Rutherford backscattering also supported the result.

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## 1. Introduction

In the information society, the demand on the further application of water is growing within the development of the next generation semiconductor devices. For example, the quest for upto-date cleaning methods that cross the limit of conventional wet processes has intensified. On the other hand, the ion beam techniques such as an ion beam etching and implantation are common and powerful techniques in the semiconductor manufacturing. Recently, another type of ion beam technique appeared, a cluster ion beam technique. Clusters are aggregates of molecules with sizes ranging from dimers to thousands of monomers. The cluster ion beam method realizes an irradiation of electromagnetically controlled ion beams that are more than one hundred times heavier than the heaviest atomic ion beam, a <sup>238</sup>U beam. The unique properties of the interaction between cluster ions and solid materials, such as a high sputtering yield and surface smoothing effect [1], are mainly attributable to a high-density energy deposition and a low-energy irradiation effect. In this article, an approach to a brand-new utilization of water, a water cluster ion beam technique is described.

# 2. Experimental procedure

Fig. 1 shows a schematic view of the water cluster ion beam apparatus. A series of four vacuum chambers form the apparatus. In the leftmost vacuum chamber in Fig. 1. a stainless steel container filled with purified water is heated by a cable heater attached to the outer wall of the container to vaporize water. A typical quantity of water is 250 mL for approximately 2 h irradiation. The temperature of the container is kept at the temperature that corresponds to the required vapor pressure  $(p_0)$  of water. For example, the temperature corresponding to the typical vapor pressure of 3 atm is 134 °C. The temperature is maintained at approximately within  $\pm 1$  °C, which corresponds to the vapor pressure variation of  $\pm 9$  kPa. The vaporized water is emitted to the vacuum chamber through a Laval nozzle. The vapor is cooled by adiabatic expansion, and clusters are formed [2]. The core portion of the stream is selected by a skimmer, and transported to the next vacuum chamber. The remaining vapor is evacuated using a roots pump. The vacuum pressures of the other three chambers are lower than the chamber containing the liquid container, and they are evacuated using diffusion pumps. The next vacuum chamber is used for differential pumping, and the neutral clusters are transported to the third vacuum chamber. In the third chamber, the neutral clusters are ionized by the electrons emitted from a loop of tungsten filament and accelerated by the voltage applied between the filament and an anode mesh. The typical voltage and current of the ionization electrons are 200 V and



<sup>\*</sup> Corresponding author. Tel.: +81 75 383 2339; fax: +81 75 383 2343.

E-mail address: ryuto@kuee.kyoto-u.ac.jp (H. Ryuto).

<sup>&</sup>lt;sup>1</sup> Formerly, H. Akiyoshi.

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Fig. 1. Schematic view of water cluster ion beam apparatus.

200 mA, respectively. The ionized cluster ions are extracted by the extraction voltage applied between the anode and an extraction electrode. The monomers and the small-sized clusters are eliminated using the retarding voltage  $(V_r)$  applied to the retarding electrode. It was reported that the velocity of the nozzle flow is highly uniform typically within 10% [2,3], so the kinetic energy of a cluster is proportional to the cluster size (the number of molecules that compose a cluster). The kinetic energy of water clusters produced at the vapor pressure of 3 atm is 0.14 eV/molecule [4]. The size-separated cluster ions are accelerated by the acceleration voltage  $(V_a)$  applied between the retarding electrode and an acceleration electrode. The typical acceleration voltage was from 3 to 9 kV. The transverse divergence of the cluster ion beam is suppressed using an Einzel lens, and the beam is transported to the sample placed in the rightmost vacuum chamber in Fig. 1. The sample and a Faraday cup were attached to a linear motion feedthrough, and one of them was placed at the peak position of the horizontal beam intensity distribution. A voltage of approximately -300 V is applied to the electron suppressor of the Faraday cup. The vertical position of the beam is fine-tuned by an electrostatic deflector. A cluster size distribution produced with a vapor pressure of 3 atm measured by the time-of-flight method has a peak approximately at the cluster size of 2500 [5]. The time variation of the beam intensity was approximately 10%. The samples were Si(100) or SiO<sub>2</sub> substrates. The sputtered depths of



**Fig. 2.** Acceleration voltage dependence of sputtered depths of Si and SiO<sub>2</sub> substrates irradiated by water cluster ion beams.

the samples were measured using a step profiler. The composite elements of the irradiated surfaces were measured by the X-ray photoelectron spectroscopy (XPS). The numbers of disordered atoms were measured by the Rutherford backscattering method of  $\alpha$  particles at 2 MeV.

## 3. Results and discussion

Fig. 2 shows the acceleration voltage dependences of the sputtered depths in silicon and silicon dioxide substrates irradiated by water cluster ion beams. The dose was  $1 \times 10^{16}$  ions/cm<sup>2</sup>. The sputtered depths both in silicon and silicon dioxide substrates increased with increase in the acceleration voltage. The sputtered depths of the silicon dioxide substrates were larger than those of the silicon substrates. The sputtering yields of the silicon and silicon dioxide substrates inradiated by 9 keV water cluster ion beams were 18 Si atoms/ion and 15 SiO<sub>2</sub> molecules/ion assuming a non-selective sputtering and densities of 2.3 and 2.2 g/cm<sup>3</sup>, respectively. Moreover, the sputtering yield of the silicon substrate irradiated by a water cluster ion beam at 9 keV was approximately 10 times larger than that irradiated by an argon monomer ion beam at the same incident energy.

Fig. 3 shows Si 2p XPS spectra of the silicon surfaces irradiated by water cluster ion beams with doses from  $1 \times 10^{13}$  to  $1 \times 10^{16}$  ions/ cm<sup>2</sup>. Two peaks appeared on the Si 2p spectrum due to chemical



Fig. 3. Si 2p XPS spectra of silicon substrates irradiated by water cluster ion beams with doses from  $1 \times 10^{13}$  to  $1 \times 10^{16}$  ions/cm<sup>2</sup>.

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