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Mass-resolved spectrometry of ion flux from hot-target reactive HiPIMS discharge with Si target

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Abstract. The work is devoted to the study of the composition of ion fluxes originating from hot-target reactive HiPIMS plasma. Experiments were carried out for thermally insulated Si target. The HiPIMS discharge was operated in O_2/Ar mixture. The component composition of ion fluxes from plasma was measured by a custom-built magnetic mass-analyzer as a function of the reactive gas flow. In the experiments, sharp changes in the ratio of fluxes of different ions were observed once the injected reactive gas flow exceeded certain levels. Nevertheless, in the HiPIMS mode, the dominant fraction of ions were those of the target material. Distinct SiO^+ signal in the ion flux was found that implies intense evaporation of oxide molecules.

Keywords: reactive magnetron sputtering, evaporation, HiPIMS, target poisoning, hot-target magnetron, copper, chromium, silicon.

1. Introduction

High-power pulsed magnetrons enable obtaining denser and stronger coatings as compared to conventional low-ionization DC magnetrons. The use of pulsed magnetron discharges in reactive modes is a promising method for obtaining optical and structural coatings of complex composition. As a rule, working with reactive gases significantly reduces the target sputtering rate and, as a consequence, the rate of coating deposition. The use of a magnetron with a hot target could overcome this drawback, but the specifics of reactive HiPIMS discharges operated with hot targets have not been studied well. Among the most valuable characteristics are the parameters of the ion fluxes ejected from discharge region that eventually arrive at the substrate surface and contribute to the coating growth.

Our work is devoted to the study of the composition of ion fluxes from hot-target reactive HiPIMS discharge. Experiments were carried out for thermally insulated Si target. These material is known for its comparatively high vapor pressure and the possibility of sustaining magnetron discharge in target vapor [1, 2]. The HiPIMS discharge was operated in O_2/Ar mixtures. The component composition of ion fluxes from plasma was measured by a custom magnetic mass-analyzer as a function of the reactive gas flow.

2. Experimental setup

The mass-resolved ion distributions were measured in a dedicated plasma characterization installation. The experimental setup scheme is demonstrated in Fig.1. The magnetron was mounted in the bottom of the vacuum chamber opposite to the mass-analyzing unit. The magnetron target was thermally insulated from the cooling system. Thermal properties of the target are discussed in [3] in detail. The deposition chamber is evacuated with a turbomolecular pump backed by a dry Roots pump to a base pressure below $2\cdot10^{-4}$ Pa. Argon and oxygen were introduced by automated mass flow controllers (Bronkhorst El-Flow series).

Mass-analyzing unit consists of a specially designed extractor (see [4] for details) and a magnetic sector. Extractor is essentially a three-electrode electrostatic lens. Its configuration sketch is also shown in Fig.1. Focusing potential was 100 V, and ion accelerating potential of 0.5 kV was applied to the second diaphragm to form the ion beam. The ion beam is deflected in the magnetic sector. Varying the magnetic field, we obtain the mass spectrum of ion beam. In order to maintain the required pressure in the mass analyzing system, it was pumped differentially.

The analyzer operation was automated and remotely controlled via wireless network. The collector was an electron multiplier VEU-6. In each case, the full mass spectrum was measured, and the peak values associated with most interesting ion species were analyzed.

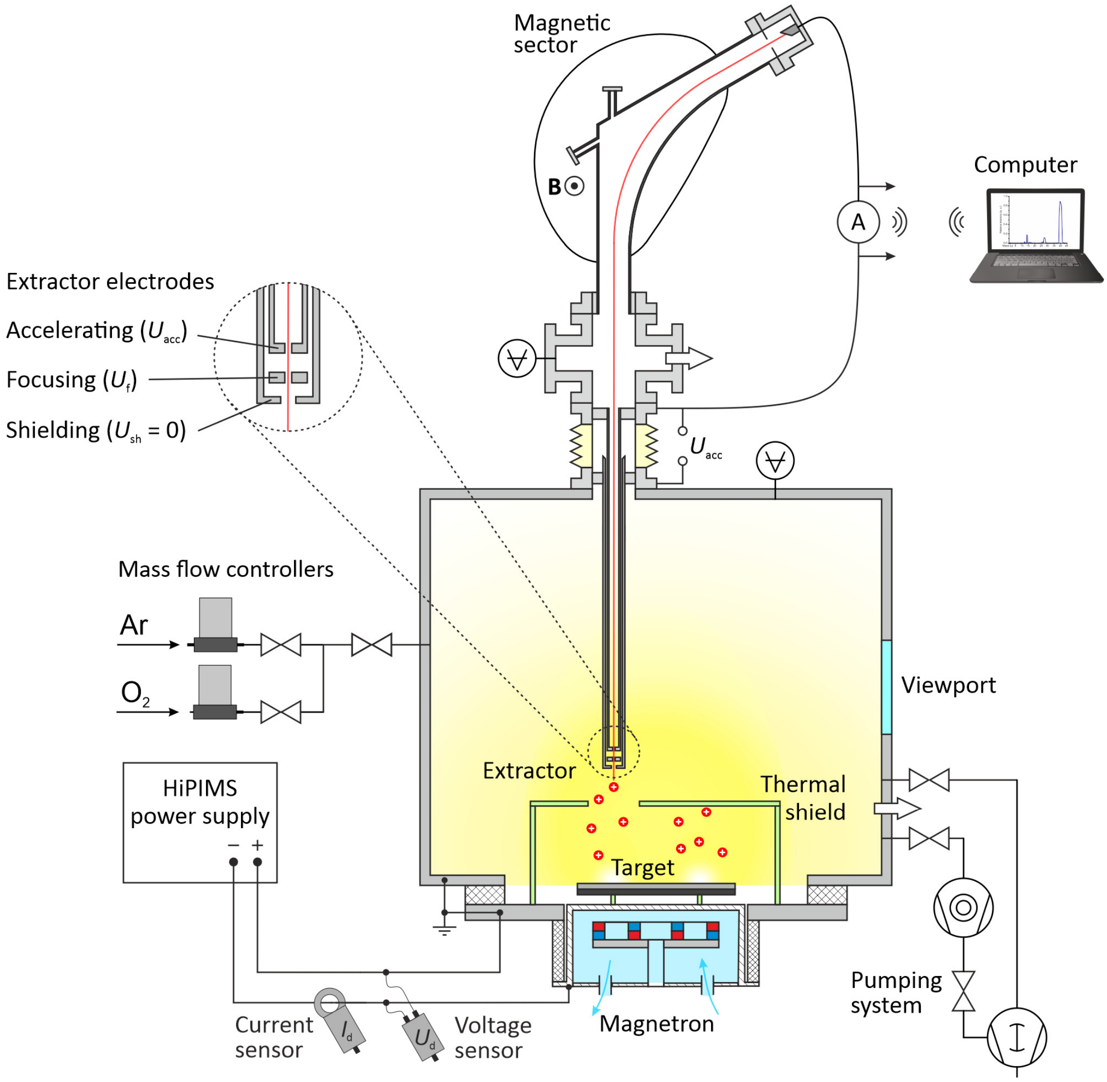

Fig.1. Experimental setup.

3. Results and discussion

All experiments were carried out for two values of the average power density of 60 and 120 W/cm^2. The power density was determined assuming that ion current is mainly concentrated in the racetrack ring, which is limited by the size of the magnets. These values are guaranteed to correspond to thermal conditions before and after the onset of active evaporation of the material with a predominance of target atoms [3]. Thus, in the 60 W/cm^2 regime, the classical process of magnetron sputtering occurs, but with a heated target, which affects the rate of chemical reactions on its surface. In the 120 W/cm^2 mode, effects associated with the evaporation of species of target material and molecules of chemical compounds (here, oxides) are added. Typical mass spectrum of ion flux from HiPIMS discharge on hot Si target (for 120 W/cm^2 mode) is shown in Fig.2.

The results of mass spectra analysis for power density levels 60 W/cm^2 and 120 W/cm^2 are shown in Fig.3 and Fig.4, respectively. Relative fractions of ions of different types in the total flux and the absolute values of the amplified current in the analyzer collector are demonstrated.

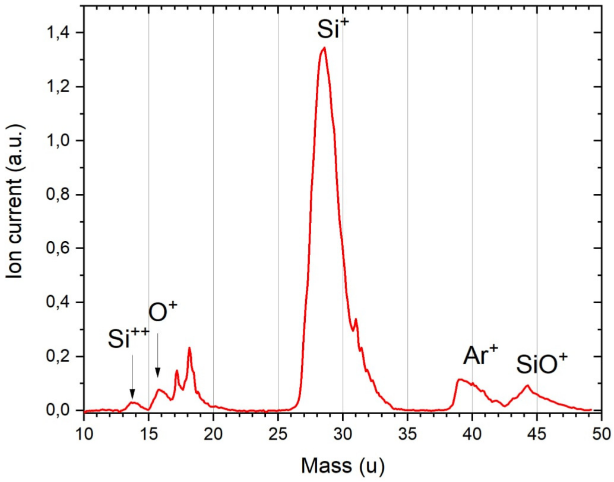

Fig.2. Typical mass spectrum of ion flux from HiPIMS discharge on Si target in O_2/Ar.

For 60 W/cm^2, as the fraction of oxygen increases, the fraction of silicon ions first gradually decreases. A sharp drop, accompanied by an increase in the fraction of molecular oxygen and argon ions, occurs at an oxygen fraction of about 25%. The visual appearance of the discharge also changes – the glow begins to fill a much larger volume and is its attachment to the target surface becomes less localized. Above a fraction of 40%, the discharge becomes unstable. When moving backwards (by decreasing the oxygen fraction), the flux of oxygen ions decreases, while that of silicon ions slowly increases and returns to its original value only when oxygen is turned off and the target is sputtered in pure argon for some time.

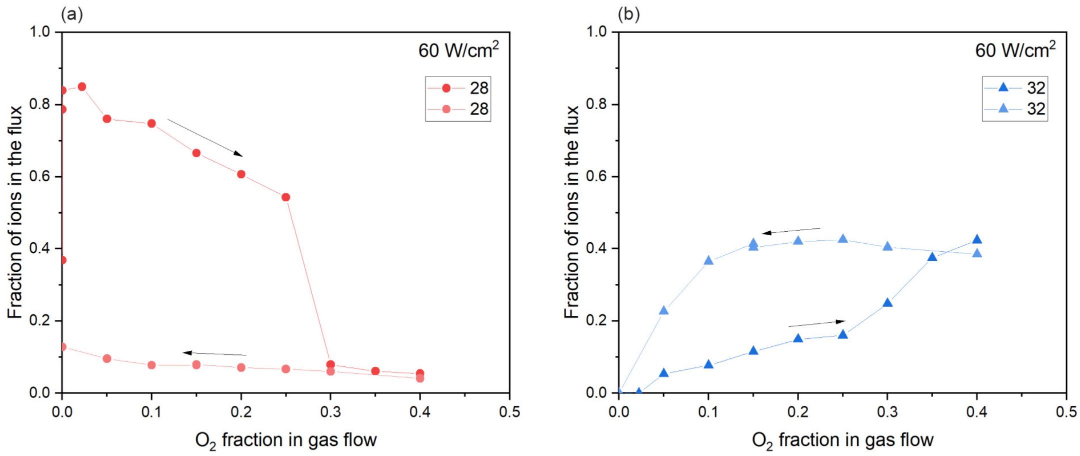

Fig.3. Dependences of the fraction of (a) Si^+ and (b) O_2^+ ions in the total flux at the substrate for a power density of 60 W/cm^2. The arrows indicate the trajectory of changes.

At a power density of 120 W/cm^2, after exceeding 30 % oxygen gas fraction, the fraction of molecular oxygen ions also begins to increase, but it does not exhibit any particularly sharp transition to the poisoned mode. A more or less rapid drop in the fraction of silicon occurs above 60% of the oxygen fraction in the gas mixture. In absolute values, when operating in 100% oxygen, the flux of O_2^+ ions is 10 times higher than that of of Si^+. In this case, flux of SiO^+ ions begin to appear, which might indicate the evaporation of silicon oxide directly in the molecular form. This observation is also indirectly confirmed by a sharp increase in the deposited mass of the coating, determined from the results of measurements by the quartz crystal microbalance (not shown).

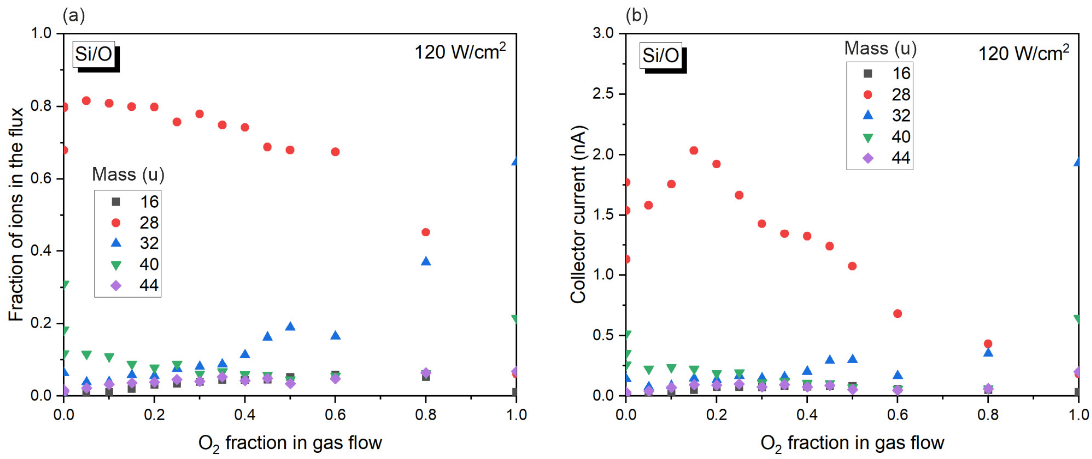

Fig.4. Dependences of (a) fractions of different ions in the total flux to the substrate and (b) amplified current in the analyzer collector for a power density of 120 W/cm^2 (Si target).

In Si/O system, thermal decomposition occurs at very high temperatures, when the saturated vapor pressure is close to atmospheric pressure, and the operation of the magnetron under such conditions is impossible. However, at temperatures above 1000°C, transformation SiO_2+Si→2SiO occurs. In this case, the pressure of SiO saturated vapor at a temperature of ~1200°C is about 1 Pa, which should have a significant effect on the behavior of the discharge.

4. Conclusion

Hot-target reactive HiPIMS magnetron sputtering of Si in Ar/O_2 mixtures is characterized by hysteresis smoothing at a power level above the start of evaporation. This should significantly increase the rate of Si_xO_y coating deposition, which is grown not only by the oxidation of the deposited silicon, but also by the deposition of the evaporated oxide. This is observed in the mass spectra of the ion flux where SiO^+ ions are clearly present.

Acknowledgements

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5. References

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