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An Introduction to Optical Emission Spectroscopy and Laser-Aided Spectroscopy Techniques for Low-Temperature Plasma Analyses

Nafarizal Nayan, Rahmat Sanudin, and Tengku Nadzlin Tengku Ibrahim

Abstract**—** Low-temperature plasma is a tool that has been used widely for surface modification of materials from 70's. Such as in the microelectronic and semiconductors industries, inductively coupled plasma and capacitively coupled plasma were used for the fabrication of thin films in ultra large scale integrated circuits. In order to optimize the plasma fabrication technique, it is important to study the plasma physics and obtain quantities like atomic, ions and electron densities, temperature of electron, atomic velocity and etc. This information is indispensable in order to control the plasma during the fabrication of thin film structure at the nano-scale level. There are several techniques of plasma diagnostics available at the moment and the amount is increasing. In the present paper, we will give a brief introduction to the optical emission spectroscopy and laser-aided spectroscopy technique for plasma studies. This is an advanced technique which allows us to study the physics inside the plasma without perturbing the plasma. The basic principles of the diagnostics will be explained and some examples from the previous experimental results will be discussed.

Keywords**:** Optical Emission Spectroscopy (OES), Laser Induced Fluorescence (LIF) Spectroscopy, Plasma Diagnostic, Atom and Ion Densities

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I. Introduction

THIS paper will give a brief introduction of optical emission spectroscopy and laser-aided spectroscopy measurement techniques for low temperature plasma analyses.

Nowadays, the plasma processing technology is vitally important for manufacturing the ultra large scale integrated circuits (ULSIs) in the electronic industries. Fabrication of ULSIs require 200~400 of steps from the production of Si substrates through final testing of a packaged components. In such steps, 40~50% of the steps are included with the plasma-aided processing technologies. In addition, the formation of metal wiring circuits and the interconnections is one of the most important steps of ULSI production.[1-3]

Basically, magnetron sputtering deposition is used to deposit metal layer during the fabrication of interconnects in ULSI. It is noted that the magnetron sputtering plasma is a low-temperature plasma that has been used widely for the formation of thin films of various applications. However, since the width and sizes of trenches and holes in ULSI shrink to below 100 nm, the conventional magnetron sputtering system become more challenging and problematic. Therefore, new concept of magnetron sputtering plasma is needed. An investigation on the plasma characteristic and properties in magnetron sputtering plasma becomes essential.

In the present paper, we will focus on the optical emission spectroscopy and laser-induced fluorescence imaging spectroscopy of magnetron sputtering plasmas. The influence of the discharge power and pressure to the plasma properties will be studied.

II. EXPERIMENTAL SETUP

OPTICAL EMISSION SPECTROSCOPY

In emission spectroscopy, energy acquired by an atom can be re-emitted as radiation which is collected and analyzed by a spectrometer. The emission of specific frequencies can be used to identify the species present in the plasma studied. From quantum theory, we know that electrons occupy discrete energy levels. Atoms, which are characterized by the energetic configurations of these electrons, emit light

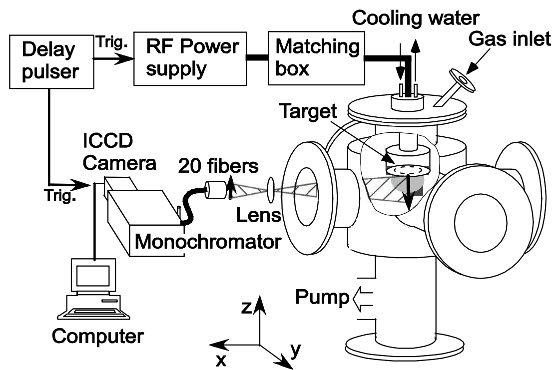

Figure 1: Experimental setup for optical emission spectroscopy.

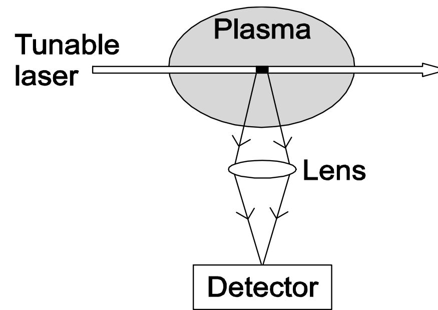

Figure 2: Experimental arrangement for laser-induced fluorescence measurement.

whenever electrons fall from a higher excited energy level to a lower level. The radiative process was originated from various reactions, such as excitation from the ground state by electron impact and de-excitation of the excited state by spontaneous emission of a photon. Therefore, numerous plasma parameters can be analyzed by means of optical emission spectroscopy (OES).

Figure 1 shows the experimental setup for optical emission spectroscopy measurement of titanium sputtering plasma. The sputtering plasma was produced by pulse-modulated rf magnetron discharge with a repetition frequency of 10 Hz and a duty ratio of 30%. A magnetron sputtering source was inserted from the top of a cylindrical chamber. The cylindrical chamber consists of four large view-ports and had a diameter of 16 cm and a length of 18 cm. The magnetron source was a conventional one with cylindrical permanent magnet on an indirect water cooling system. The strength of the magnetic field on the cylindrical axis was the maximum value of 120 mT on the target. The titanium (Ti) target with a diameter of 5 cm was used. Base pressure of the vacuum chamber was approximately 10^{-7} Torr, which was obtained by evacuating the chamber using the combination of a turbomolecular and oil rotary pumps. The gas pressure was controlled using a gate valve near the pumping unit and it was measured using the capacitance manometer.

As shown in Fig. 1, the optical emission image along the cylindrical axis of the discharge was projected onto an optical fiber array using an imaging lens. The optical fiber array was composed of linearly-aligned 20 cores, and was placed to observe the axial distribution of the optical emission. The other end of the optical fiber array was placed closed to the entrance slit of a monochromator with a focal length of 500 mm. The exit side of the monochromator was connected to an ICCD camera, by which we recorded optical emission spectra at various distances from the target surface.

LASER-INDUCED FLUORESCENE IMAGING SPECTROSCOPY

Laser-induced fluorescence (LIF) is an advanced technique in measuring the spatial distribution of particles in plasma. The advantage of LIF technique is that LIF provides a sensitive detection of particles in plasmas without perturbing the plasma properties. In addition, LIF enables us a measurement of high accuracy and high temporal and spatial resolutions.

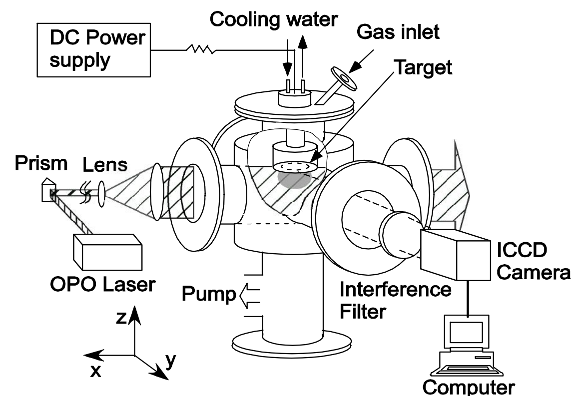

Figure 3: Experimental setup for two-dimensional laser-induced fluorescence imaging spectroscopy.

Figure 2 shows the experimental arrangement for LIF measurement system. A tunable laser beam is injected into the plasma and excites the particles from their ground state to upper state at their resonant wavelength. Then, when the particles at excited state transfer to lower state, fluorescence light will be emitted by means of energy level transition. LIF emission is collected by an optical system and a detector. The LIF emission intensity is proportional to the density of particles at their ground state. In the present work, we applied the two-dimensional LIF measurement in which can determine two-dimensional spatial density distribution of atoms and ions in plasmas.

Figure 3 shows the experimental setup for two-dimensional laser-induced fluorescence imaging spectroscopy measurement.[4,5] Tunable laser pulses yielded from an optical parametric oscillator (OPO, Spectra-Physics) were injected into plasmas in front of the Ti target. The tunable laser beam was arranged to have a planar shape by using two cylindrical lenses. The width and the thickness of the planar beam were 80 and 2 mm, respectively. The wavelength of the OPO laser was tuned to excite Ti atoms and Ti^+ ions in plasmas at their resonant wavelength. Excited Ti and Ti^+ yielded fluorescence on the planar laser beam, and the pictures of the fluorescence images were taken by a gated charge-coupled device (CCD) camera with an image intensifier (ICCD: Princeton Instruments). The gate width of the ICCD camera was adjusted (approximately tens to

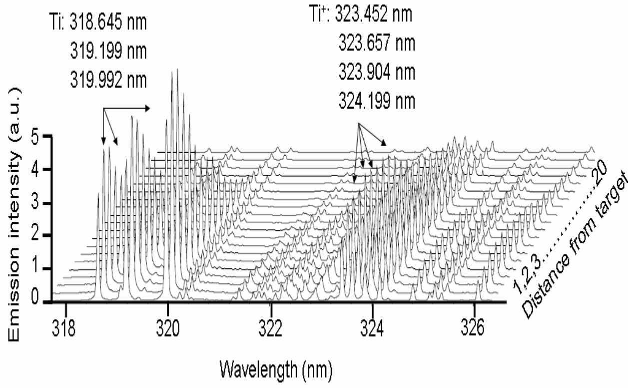

Figure 4: Raw data of the emission spectrum from Ti sputtering plasma.

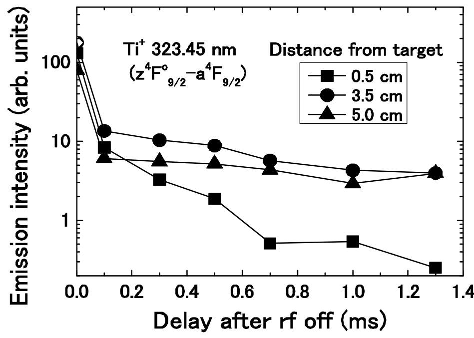

Figure 5: Temporal variations of excited Ti^+ at various distances from target.

hundreds ns) depends on the LIF emission lifetime, so that the LIF intensity was maximum.

The tones of the fluorescence images represented the two dimensional distributions of the Ti and Ti^+ densities at their initial states. Interference filters were placed to separate the fluorescence from the stray lights and self-emissions from plasmas. In this way the two-dimensional distributions of the Ti and Ti^+ densities in the plasmas were obtained. The influence of the slight distribution of the laser intensity on the modulation of the LIF intensity was negligible since the laser intensity was strong enough to saturate the LIF intensity.

III. RESULTS AND DISCUSSIONS

RADIATIVE TI AND TI+ DENSITIES

Figure 4 shows the emission spectrum from Ti sputtering plasma in the range from 318 to 326 nm. The rf power and the Ar pressure were 100 W and 150 mTorr, respectively. Since the fiber detector is composes of 20 linearly-aligned cores, the results shows the emission spectrum at various distance from the Ti target.

Figure 4 shows that the optical spectrum from radiative Ti and Ti^+ is observed clearly at this region. The emission intensity of Ti and Ti^+ decreased rapidly with the distance from the target. This is easily understandable since the radiative Ti and Ti^+ are originated from the electron impact

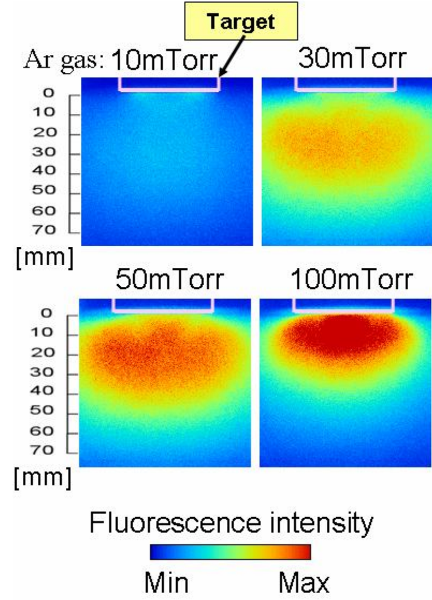

Figure 6: Two-dimensional LIF images of Ti density at various Ar pressure. The dc power was fixed at 75 W.

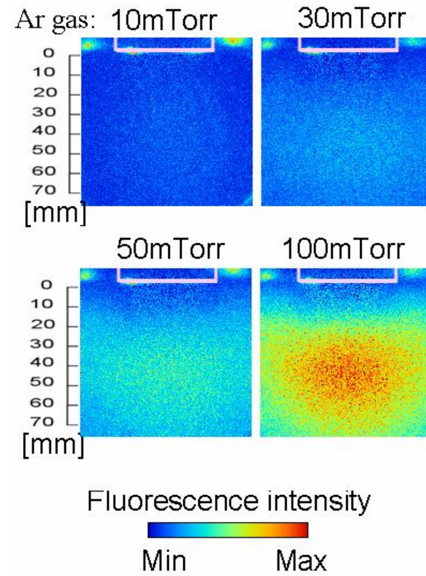

Figure 7: Two-dimensional LIF images of Ti^+ density at various Ar pressure. The dc power was fixed at 75 W.

excitation. The hot electron region is located at the vicinity of the Ti target due to the ***E***×***B*** effect at this region.

By changing the delay time of the ICCD camera from the termination of the rf power, the temporal variation of the spatial distributions of Ti and Ti^+ densities in the afterglow were measured. Due to the limitation of page, the result of the temporal variation of radiative Ti will be shown at the conference.

Figure 5 shows the optical emission intensities from Ti^+ at various distances from the target as a function of time after the termination of the rf power. As shown in Fig. 5, the lifetime of the radiative Ti^+ was long, where the optical emission intensities from Ti^+ at 3.5 and 5 cm from the target were almost constant between 0.2 and 1.2 ms.

GROUND STATE TI AND TI^+ DENSITIES

The density distribution of Ti and Ti^+ were measured at various dc power and gas pressures. As a result, the amount of Ti and Ti^+ increased with the dc power and the gas pressure. In this paper, the experimental results observed at relatively high gas pressures will be emphasized.

Figure 6 shows the two-dimensional images of the Ti densities in a region of 7cm×7cm in front of the magnetron source. The dc power was fixed at 75 W, and the measurement was repeated at four gas pressures of 10, 30, 50 and 100 mTorr. As shown in Fig.6, it was found that the peak of the Ti density became closer to the target at a higher gas pressure. These results are attributed to the larger number of sputtered particles and the less efficient diffusion in a higher-pressure discharge.

Figure 7 shows the spatial distribution of Ti^+ densities at the same discharge conditions. In contrast to Ti density, as shown in Fig.7, it was found that the peak of the Ti^+ density was separated from the target suface. There are no reasonable explanations for the peak position at the moment, but this result suggests that there may be a production source of Ti^+ in the gas phase. In addition, it was observed that the Ti^+ density increased drastically with the gas pressure.

In the magnetron sputtering plasma source, the bright plasma was localized in the adjacent region to the target surface. The experimental result shown in Fig. 6 and 7 suggest that ionization processes are available in the dark plasma at a distance of several centimetres from the target. A possible ionization process in the dark plasma is collision with Ar at metastable state (Penning ionization). However, our experimental results on Ti^+ and Ar^M were not consistent with the assumption of Penning ionization. Further investigations on this subject are necessary.

IV. CONCLUSION

The spatial and temporal variation of radiative Ti and Ti^+ were successfully measured using optical emission spectroscopy technique. The distribution of radiative species as a function of distance from the target surface and as a function of time after the termination of the rf power gave us deep understanding on the plasma processes occurred in the sputtering plasma.

In addition, the ground state densities of Ti and Ti^+ were successfully measured using the laser-induced fluorescence imaging spectroscopy technique. The results show that the ground state densities of Ti and Ti^+ were changed drastically with the discharge gas pressure. This result was useful in order to understand the mechanism of ionization in high-pressure magnetron sputtering plasmas.

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