

# Laser-Aided Diagnostics of Atoms and Particulates in Magnetron Sputtering Plasmas

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**Abstract ?** There has been much interest recently in developing reflectarray Laser-aided diagnostic technique is introduced as an advanced and valuable technique to evaluate the properties of plasma. This technique is an expensive and sophisticated technique which requires researchers to have a basic knowledge in optical spectroscopy. In the present paper, we will generally introduce the experimental work using laser-induced fluorescence (LIF) and laser light scattering (LLS) techniques. The LIF was used to evaluate the spatial distribution of Cu atoms in magnetron sputtering plasma. The change in the spatial distribution was studied as a function of discharge power. On the other hand, the LLS was used to evaluate the generation of Cu particulates in high-pressure magnetron sputtering plasma. The temporal evolution of Cu particulates in the gas phase of sputtering plasma was visualized successfully.

**Keywords:** *Laser Induced Fluorescence (LIF), Atomic and Ion Densities, Laser Light Scattering, Particulate*

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**A**t present various types of diagnostic techniques are being used for the study of plasmas, such as Langmuir probe measurement, mass spectrometry, optical spectroscopy, and laser-aided spectroscopy. In general, each technique has its own strength and weakness. For example, the Langmuir probe measurement technique is a widely used diagnostic for glow discharge plasma to measure the plasma potential and electron density. However, the Langmuir probe cannot determine the neutral particle density of the plasma, which is the most important parameter during the deposition of thin film.

The optical and laser-aided spectroscopy is a technique to measure the neutral species (atoms or molecules) in the plasma without perturbing the plasma physically. In the present paper, the laser-aided spectroscopy will be introduced briefly.

### LASER-INDUCED FLUORESCENCE SPECTROSCOPY

Laser-induced fluorescence (LIF) is an advanced technique for measuring the spatial density distribution of neutral particles. The advantage of LIF technique is that it provides a sensitive detection of particles in plasmas with high accuracy and high temporal and spatial resolutions without perturbing the plasma properties.

Figure 1 shows an experimental arrangement for LIF measurement of plasma. First, a tunable laser beam is injected into the plasma and excites the atoms from their ground state to upper state at their resonant wavelength. When the particles at excited state decay to lower state, fluorescence light will be emitted. The emitted fluorescence is called laser-induced fluorescence. The LIF emission is collected by an optical system and a detector such as monochromator or ICCD camera. The LIF emission intensity is proportional to the density of particles at their ground state before excitation. Hence, high sensitivity of spatial measurement can be achieved if we change the coordinate of optical system, detector and laser beam.

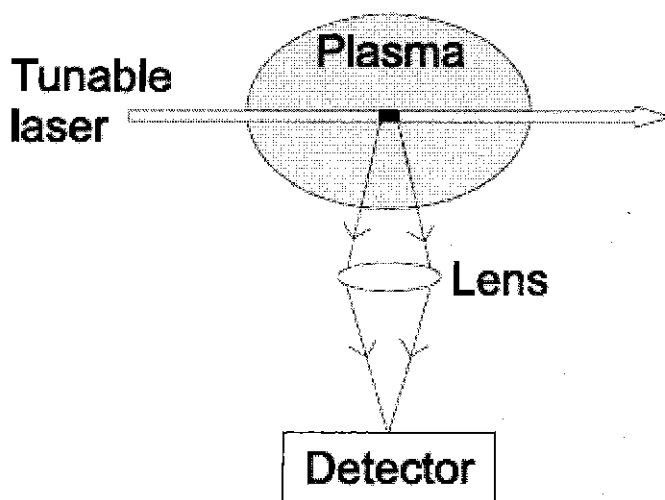


FIGURE 1. Arrangement for LIF measurement.

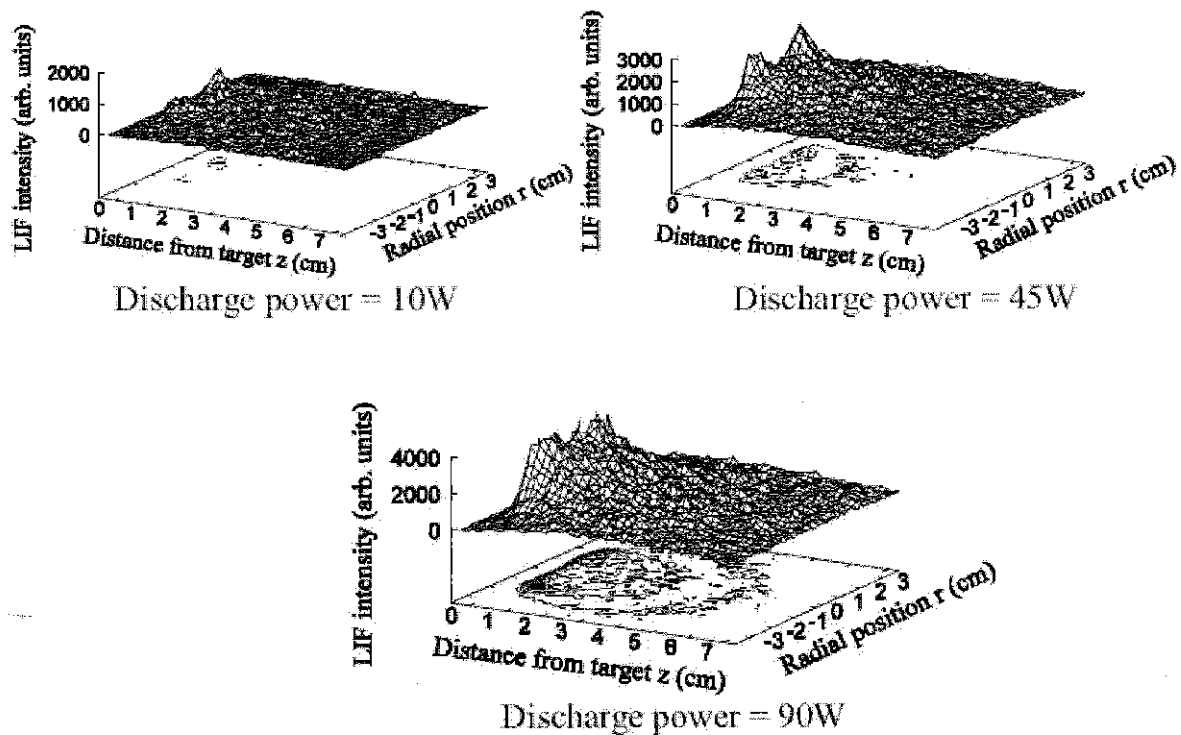
As an example of the LIF technique, we describe the two-dimensional (2-D) LIF measurement in which the two-dimensional spatial density distribution of atoms in a Cu magnetron sputtering plasma can be determined. The experimental apparatus for 2D-LIF measurement had been described elsewhere [1]. Briefly, a tunable laser pulse produced by an optical parametric oscillator (OPO) laser (Spectra-Physics, MOPO-730, FDO-900) is injected into the plasma in front of the target. The repetition rate of the OPO laser is 10 Hz and the spectral line width is  $0.25 \text{ cm}^{-1}$ . The laser output energy is set at the range of 3~6 mJ depending on the wavelength used. The OPO laser beam is arranged to have a planar shape by using two cylindrical lenses. The width and the thickness of the planar OPO laser beam are 80 mm and 2 mm, respectively.

The wave length of the OPO laser is tuned to excite the atoms and ions in the plasma. The fluorescence emission from the excited atoms and ions is detected by a gated charge-coupled device (CCD) camera with an image intensifier (ICCD: Princeton Instruments, PI-MAX-512RB). The camera's gain value is set at 200 and the gate width and accumulation shots of the ICCD camera are 100 ns and 200 times respectively for all experiment conditions unless there are changes in the experimental set-up. It should be note that the gate width (opening time) should be set to cover the OPO pulse width of near 10 ns.

The magnetron sputtering discharges are produced by a direct-current power supply with Cu target of purity 99.96% and diameter of 50 mm. The spatial density distribution of Cu for various discharge power and gas pressure are measured. For the LIF measurements, the tunable OPO laser is tuned to 324.750 nm to excite the Cu atoms from  $s^2S_{1/2}$  state to  $p^2P^o_{3/2}$  state. [2] The laser output energy is approximately 3 mJ as measured at the diagnostic port of the vacuum chamber which is approximately 30 cm from the symmetrical axis of Cu target. Since we arrange the OPO laser beam to a planar shape of 80 mm width and 2 mm thickness using two cylindrical lenses, the fluence of OPO laser is approximately  $1.9 \text{ mJ/cm}^2$ . The induced fluorescence at 510.550 nm from  $p^2 P^o_{3/2}$  state to  $s^2 D_{5/2}$  state is monitored using a gated ICCD camera.

Figure 2 shows the 3-dimensional graphs of 2D-LIF images observed by ICCD camera. The z and r axes refer to the distance from the target surface and the radial position from the symmetrical axis of target, respectively. The vertical axis refers to the LIF intensity recorded by the ICCD camera. It shows the density distribution of Cu atoms at various discharge powers. The Ar gas pressure is fixed at 5 mTorr. At low discharge power of 10 W, the Cu density is low and almost undetectable. However, when the discharge power is increased we can see that the Cu density increases. At high discharge power of 90 W, the relative peak density of Cu is 4 times higher than that at discharge power of 10 W. Basically, the increase of Cu atom density with discharge power is due to the increase of Ar+ ions energy which sputters out more Cu atoms from the target.

At discharge power of 45 W, we can see two peaks of Cu density clearly. This is because the electrons are trapped at this region by  $E \times B$  effect and thus enhances the ionization of the Ar atoms, consequently enhanced sputtering. The results indicate that the spatial distribution of Cu atoms is accurately visualized from LIF measurement.



**FIGURE 2.** Density distribution of Cu atoms at various discharge powers. The Ar gas pressure was 5mTorr.

### LASER LIGHT SCATTERING TECHNIQUE

The laser light scattering technique has been carried out to visualize the particulates in plasmas by many researchers in Japan and Europe.[3] The laser beam is expanded into the plasmas and the laser light scattering intensities from particulates are measured. The laser light scattering intensity is a complex function of particle size, size distribution, shape, and refractive index. In addition, the number density is also expressed in terms of directions, wavelengths and polarization of the incident and scattering light. Thus, sophisticated methods are needed to determine the particle properties.

Shiratani *et al.* have developed a two-dimensional polarization-sensitive laser light scattering method to measure the profile of size and density of particles in RF silane plasmas. [3] Briefly, in this method, two laser beams with different wavelength are polarized horizontally and vertically to the observation plane and injected into the RF silane plasmas. The particle size and density in the plasma can be deduced using the scattering intensity ratio of both wavelengths. Such method provides an in situ measurement of the particle size and density with good time and spatial resolution.

The laser light scattering technique has been applied to visualize the generation of Cu particulates in magnetron sputtering plasmas. The experiments are done using only one wavelength and the laser beam is not polarized. The laser light scattering intensities obtained in the present experiments have no information of particulate size and density. However, it should be noted that from Mie scattering theory light scattering only occurs when the particles size is the same or longer than the illuminating laser wavelength. As an example of this technique, we describe an experiment to measure the spatial evolution of

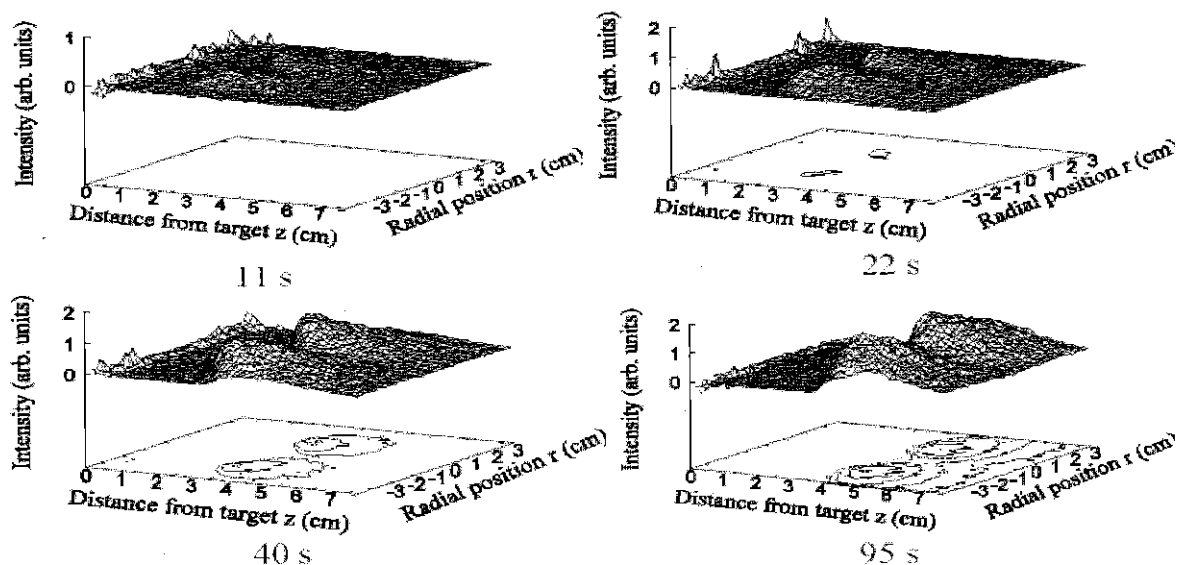
particulates in gas phase of magnetron sputtering plasmas. The laser wavelength used in this experiment is 496 nm.[4]

The experimental apparatus is similar to that used for 2D-LIF measurement, where the magnetron sputtering plasma is produced by a dc discharge using a copper target. Ar gas is not introduced from the top of vacuum chamber as that of LIF measurement, but from a diagnostic port far from the magnetron source. The OPO laser beam of wavelength 496 nm is arranged to have a planar shape and was injected into the plasma. The laser energy used is approximately 45 mJ.

An ICCD camera is set to determine the laser light scattering at angular position of  $90^\circ$  to the laser beam. The interference filter of  $496 \pm 5$  nm is used to separate the scattered light from the plasma emitted light. The camera's gain value is set at 200 and the gate width (exposure time) is 200 ns. The images are accumulated for 10 exposures to have an enhanced image of the scattered light.

The two-dimensional laser light scattering images are taken starting from the instant of plasma ignition and it covered a time range of several minutes. Figure 3 shows the 3-D graph of the temporal variation of laser light scattering. The dc power and gas pressure are fixed at 4 W and 200 mTorr, respectively. From the scattering images in Fig. 3, we found that at 11 s, a low intensity of scattered light is visualized at the boundary region of the plasma. The intensity increases with the discharge running time. Therefore, we may conclude that during the time from 0 to almost 1 minute after the plasma initiation the particulates start to grow and this growth reaches saturation phase after 1 minute. The coagulation between the particles is responsible for the particulates growth.

On the other hand, at 95 s, we can see that the two locations with peak intensity are at the downstream region. This indicates that there is a force which accelerates the particulates toward the chamber wall. The force may become more crucial as the particulates size increases.



**FIGURE 3:** Temporal evolution of Cu particulates in magnetron sputtering plasma. The dc power was 4 W and the gas pressure was 200 mTorr.

## CONCLUSIONS

The spatial distribution of atomic density was successfully observed using 2D laser-induced fluorescence imaging spectroscopy technique. The results are useful in order to understand the deposition mechanism using sputtering plasma. On the other hand, the laser light scattering is also successfully used to evaluate the temporal evolution of Cu particulates growth in the sputtering plasma.

## ACKNOWLEDGMENT

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