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An rf sustained argon and copper plasma for ionized physical vapor deposition of copper

W. Wang, J. Foster, T. Snodgrass, A. E. Wendt, and J. H. Booske
Engineering Research Center (ERC) for Plasma-Aided Manufacturing, 1410 Engineering Drive, University of Wisconsin-Madison, Madison, Wisconsin 53706

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Langmuir probe, optical emission spectroscopy, and biased quartz crystal microbalance measurements were used to investigate an argon and copper plasma used for ionized physical vapor deposition of copper. Copper vapor generated by a magnetron sputter discharge is ionized upon passing through an argon discharge excited by an internal rf induction antenna. Argon plasma characteristics such as electron temperatures T_e, plasma densities n_e, and plasma and floating potentials V_p and V_f, were studied as a function of argon pressure and rf power. An increase of plasma density versus rf discharge power and argon pressure was observed. The radial profile of plasma density measured by a Langmuir probe reveals a peak ion density at the center of the rf antenna and an increase in the radial ion concentration gradient with argon pressure. The ratios of optical emission intensities from Cu^+ ion and Cu neutral lines increase with rf discharge power and argon pressure. The biased quartz crystal microbalance measurements show an increase of both Cu^+ ion flux and the ratio of Cu^+ ion to Cu neutral fluxes with rf power and argon pressure; however, they also show a decrease of total Cu flux with increasing argon pressure. © *1999 American Institute of Physics.* [S0021-8979(99)00811-7]

I. INTRODUCTION

The shrinking of semiconductor device size to the subquarter micron regime places stringent demands for metals and metal alloys film deposition technology for contact and interconnect applications for ultralarge scale integrated (ULSI) circuit fabrication. Conventional sputtered physical vapor deposition (PVD) becomes insufficient to fill trenches of high aspect ratio with metals because of pinch off at the top of the trench and void formation inside the trench. Similar geometric shadowing effects make it difficult to obtain uniform, conformal, well-adhered, thin seed films with conventional PVD in high-aspect-ratio trenches and vias. Recently, ionized physical vapor deposition (IPVD) has been demonstrated as an improvement to conventional PVD for depositing metal films into deep trenches of high aspect ratio in semiconductor device manufacturing.[1–4]

In ionized sputtering, a high density argon plasma (10^{11}–10^{12} ions/cm^3) is produced between the sputtering target and the wafer pedestal by using a rf inductively coupled plasma source. When the sputtered metal atoms traverse this high density plasma region, they may be ionized. Because of its lower ionization energy for metal atoms (~8 eV) compared to argon (~16 eV), a larger fraction of the sputtered atoms may be ionized. By applying a negative voltage to the substrate, the positive metal ions are accelerated toward the substrate forming metal films. Guided by the electric fields in the plasma sheath above the substrate, the metal ions can be directed into high aspect ratio trenches, forming improved films due to the ions' greater directionality compared to metal neutrals in normal sputtering deposition. At the same time, resputtering from these energetic ions further reduces the pinch off at the top of the trench and leads to better coverage on the side walls of trenches.[5] This resputtering pheonomenon is also perceived to be important for realizing uniform conformal seed films in high-aspect-ratio features.[4]

The ionization of sputtered atoms for ionized sputtering in an inductively coupled rf argon plasma is influenced by plasma density n_e, electron temperature T_e, and argon pressure P. Plasma conditions determine the ion and neutral fluxes and ultimately the film properties in ionized sputtering. In this study, these plasma characteristics were investigated as functions of rf power and argon pressure by using a Langmuir probe. The probe was also used to measure the radial plasma density profile at different argon pressures.

Copper ionization trends were monitored using optical emission spectroscopy and a biased quartz crystal microbalance. The emission intensities from Cu^+ ion (213.5 nm) and Cu neutral (216.5 nm) lines were recorded as functions of rf power and argon pressure.[6] Changes in the line emission intensity ratio of Cu^+ to Cu in an argon plasma are proportional to changes in the Cu^+ to Cu density ratio for conditions of constant electron temperature. Hence, such a study provides information on the degree of Cu ionization in argon plasmas, under constant electron temperature conditions.

The Cu neutral, Cu^+ ion fluxes, and Cu ion flux fraction, defined as the Cu^+ ion flux divided by the sum of the Cu neutral and Cu^+ ion fluxes, were measured by a biased microbalance at different rf powers and argon pressures. The microbalance was configured such that a dc bias can be directly applied to the quartz crystal without using a metal grid.[7] This configuration eliminated the inaccuracies associated with using an additional biased grid in front of the microbalance to repel positive Cu^+ ions.[2]

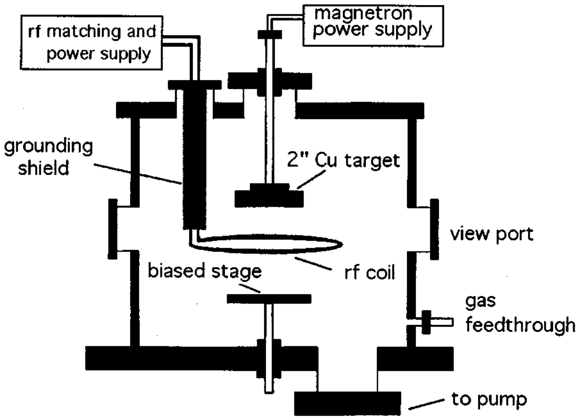

FIG. 1. A schematic drawing of the experimental apparatus for ionized sputtering of Cu.

II. EXPERIMENT

Figure 1 represents a schematic drawing of the ionized sputtering system for this study. It is slightly different from the previously reported ionized sputtering system where a multipole confinement magnetic field was applied to enhance the plasma density.[8] The system consists of an Al vacuum chamber 45 cm in diameter and 50 cm in height. The chamber was evacuated with a turbomolecular pump to a base pressure of 1×10^{-6} Torr. An rf antenna of Cu tubing [outer diameter (OD) 0.635 cm] was installed from the top of the chamber and was water cooled during the experiment. A grounded metal shield was used to eliminate plasma generation between the two leads. The single turn rf induction antenna, which had a diameter of 18 cm, was connected to a capacitive matching network placed on top of the chamber. A dc magnetron that utilized a Cu target of 5 cm diameter served as the source of Cu metal vapor. The sputtering target of 99.995% was located 5 cm above the center of the rf antenna and the wafer stage was located about 12 cm away from the target.

The electron temperatures, ion densities, and plasma potentials of rf argon plasma were determined by using a cylindrical Langmuir probe with a computer data-acquisition system. About 40 current–voltage ($I-V$) traces were ensemble averaged for each probe measurement. The probe-driver circuitry provides a voltage in the range of ±125 V and is controlled by the computer via a 24-bit digital-to-analog converter. The probe current is determined by passing the current through the current-sensing resistor, which is chosen to produce a ±10 V signal at a full-scale current. The ion density was determined from the ion saturation region in the $I-V$ curve using a square-root model.[9] The electron temperature was determined from the inverse slope of the logarithmic electron current, and the plasma potential is identified as the probe voltage at which the second derivative of the $I-V$ trace passes through zero.

The probe consists of a tungsten (W) wire tip of 8 mm long and 0.2 mm diameter. A recessed structure was employed on the top of the probe so the effect of metal contamination from the antenna sputtering can be minimized. The probe tip was placed in the center of the chamber and 5 cm below the rf antenna. A tuned resonant rf filter in combination with a low-pass filter was used to maximize the probe impedance and to minimize the rf distortion to the probe measurements.[10,11] All the Langmuir probe measurements were conducted in an argon plasma while the magnetron was off. Argon was assumed as the only species present in the plasma. This was based on the observation that the Cu sputtering from the antenna by the rf self-bias voltage was small and therefore was ignored.

The optical emission spectra acquired from the Ar/Cu plasma were recorded by using a 0.5 M scanning spectrometer with a Princeton Applied Research photodiode array as a detector and 1200 g/mm grating. The detector was cooled at −25 °C and purged with dry nitrogen during the measurements. The emission was collected by a 2.5 mm optical fiber through a 3 cm diam quartz window located 2 cm below the center of the chamber. The intensity ratio of Cu^+ ion at 213.5 nm and Cu neutral at 216.5 nm was used as an indication of changes in the degree of Cu ionization in the Ar/Cu plasma.[6]

Cu neutral, Cu^+ ion fluxes and the ion flux fractions were measured by using a biased quartz crystal microbalance. Details about the construction of the biased microbalance is described in Ref. 7. Briefly, the biased microbalance was constructed from a commercial thin film deposition rate monitor. An additional electrical connection to the quartz crystal was made, so that a dc bias could be directly applied to the face of the quartz crystal. Without a bias, both Cu neutral and Cu^+ ion fluxes (total flux) are measured. When a positive bias voltage above the plasma potential is applied, Cu^+ ions are repelled from the microbalance and only the Cu neutral flux is measured. The Cu^+ ion flux is determined by subtracting the Cu neutral flux from the total flux for Cu^+ ion and Cu neutral.

III. EXPERIMENTAL RESULTS

A. Langmuir probe measurement

The electron temperature and plasma density of the rf argon plasma determine the Cu ionization probability in ionized sputtering. In particular, the electron impact ionization rate constant for Cu increases with electron temperature. Figure 2 shows the electron temperature T_e and the difference between plasma and floating potentials $V_p'-V_f$ as a function of the argon pressure at a rf discharge power of 400 W. The electron temperature decreases with argon pressure from 4.5 eV at a pressure of 1 mTorr to about 1.8 eV at 60 mTorr. Figure 2 also shows that the quantity of (V_p-V_f) decreases in proportion with the electron temperature. This behavior is expected for a Maxwellian electron population. The plasma-to-floating potential differences are between 7 and 10 V at argon pressures of 20–40 mTorr.

The plasma densities versus rf discharge power at argon pressures of 10, 20, and 40 mTorr are shown in Fig. 3. The densities increase with rf power as expected. Similarly, as expected, they are larger at higher argon pressures with the same rf discharge power. The argon plasma density increases

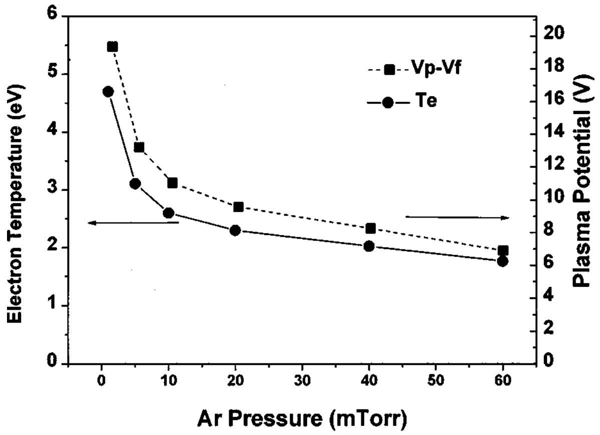

FIG. 2. Electron temperatures and plasma-to-floating potential differences in a rf argon plasma vs the argon pressure. The discharge was excited at a rf power of 400 W.

to 5×10^{11} ions/cm^3 at an rf discharge power of 600 W and argon pressure of 40 mTorr.

Figure 4 represents the ion saturation currents at various radial locations and at argon pressures of 10, 20, and 40 mTorr, respectively. The ion saturation current was measured by using a Langmuir probe which was biased with a negative dc voltage of -50 V. A rf discharge power of 400 W was used for these measurements. As the plasma density is proportional to the ion saturation current, this measurement illustrates the radial plasma ion density distribution for an internal rf antenna. At low pressure of 10 mTorr, the ion saturation current is smaller due to a lower plasma density. It shows a peak intensity at zero on the x axis corresponding to the center of the antenna and decreases along the radial direction. As the argon pressure increases, the ion saturation current peak value increases because of a higher plasma density at higher pressure. However, the radial gradient for ion density also increases with pressure, leading to a less uniform plasma for radii smaller than that of the antenna at high pressures.

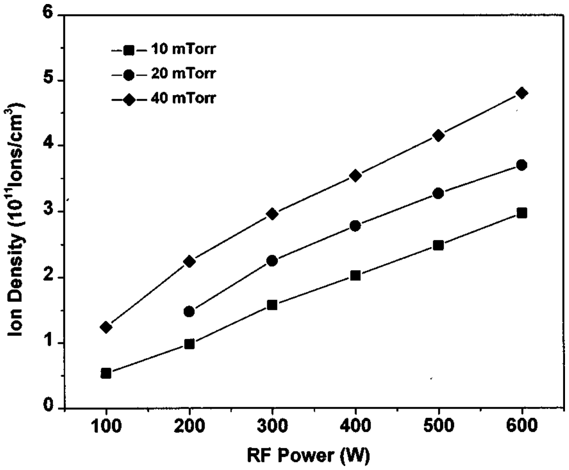

FIG. 3. The ion densities at argon pressures of 10, 20, and 40 mTorr vs the rf power.

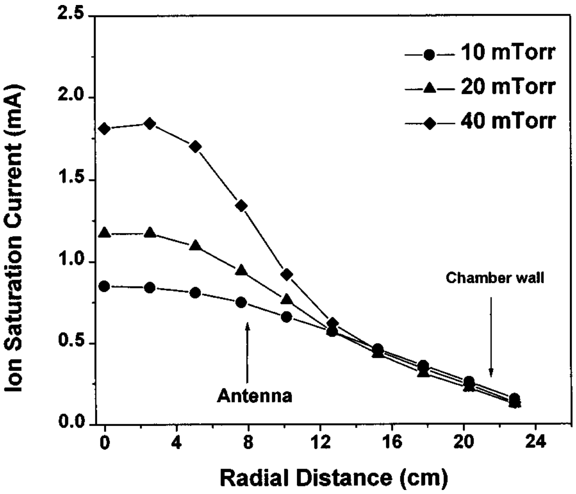

FIG. 4. Ion saturation currents at 10, 20, and 40 mTorr as a function of radial location.

B. Optical emission spectroscopy

Assuming electron impact excitation from the ground state to the exciting state, the optical emission intensity for Cu neutrals and Cu$^+$ ions in an argon plasma is

$$I=n\int_0^\infty \sigma v f(E)dE=nk(T_e), \tag{1}$$

where σ is the velocity-dependent cross section for electron impact excitation of the Cu atom or Cu$^+$ ion, $f(E)$ is the electron energy distribution function for argon plasma, v is the electron velocity, E is the electron energy, and n represents the Cu neutral or Cu$^+$ ion density.[6] For a Maxwellian distribution of electron energies, the integral in Eq. (1) is a function of electron temperature represented by an electron temperature dependent rate constant $k(T_e)$.[12] This shows that the emission intensities from both Cu neutrals and Cu$^+$ ions are proportional to the density of the species. The constant of proportionality is the same for plasmas with the same electron temperature. The ratio of the emission intensities from Cu neutral and Cu$^+$ ion lines at a constant argon pressure will be proportional to the degree of Cu ionization

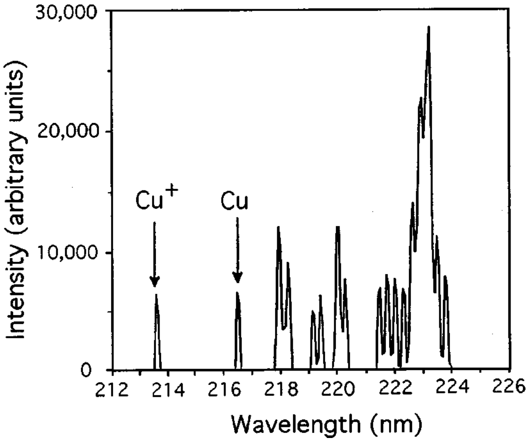

FIG. 5. An optical emission spectrum from Ar/Cu plasma at wavelength between 212 and 226 nm.

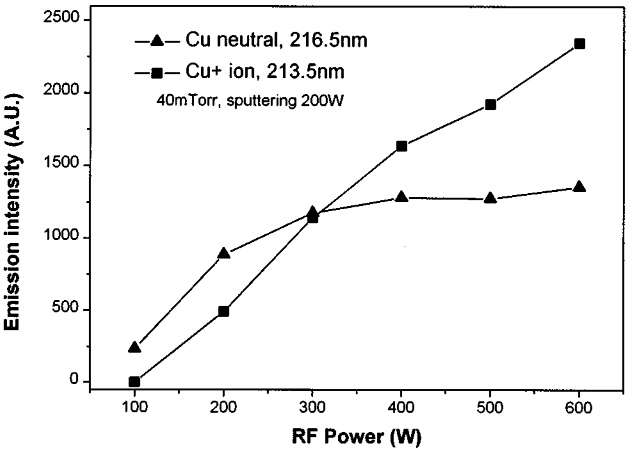

FIG. 6. The optical emission intensities from Cu neutral and Cu$^+$ ion lines at 40 mTorr vs rf discharge power.

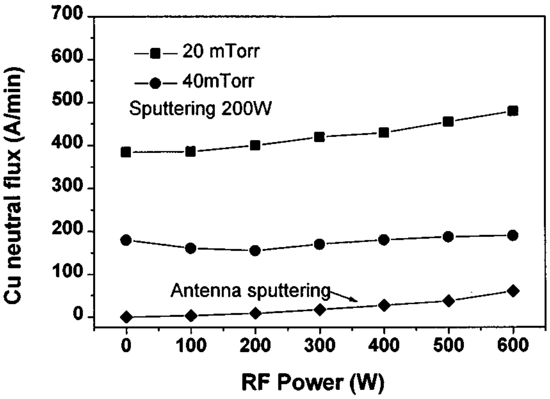

FIG. 8. The Cu neutral deposition fluxes at 20 and 40 mTorr as a function of rf power. The magnetron sputter power is 200 W. Also shown is deposition rate due to antenna sputtering with 0 W magnetron sputter power and 20 mTorr Ar pressure.

as rf power is varied in an argon plasma, because the electron temperature is mainly independent of rf power.

The optical emission spectroscopy of Ar/Cu plasma was performed as a function of rf power and at argon pressures of 10, 20, and 40 mTorr, respectively. A typical emission spectrum of wavelength between 212 and 226 nm from the Cu/Ar plasma is shown in Fig. 5. This spectrum was recorded at an argon pressure of 20 mTorr, rf power of 400 W, and Cu sputtering power of 200 W. The two emission peaks at 216.5 and 213.5 nm as indicated in the spectrum represent the emissions from the Cu neutral and Cu$^+$ ion lines, respectively. Their peak intensities and the ratio were recorded as ionization trends at various rf powers and argon pressures in Figs. 6 and 7.

Figure 6 compares the emission peak intensities from Cu neutral and Cu$^+$ ion lines versus the rf power at an argon pressure of 40 mTorr. A Cu sputtering power of 200 W was used. The emission intensities from Cu neutral and Cu$^+$ ion lines both increase with the rf power. The emission intensity from the Cu$^+$ ion line increases faster at higher rf power compared to that of the neutral Cu line.

The emission intensity ratios from the Cu$^+$ ion line and Cu neutral line at argon pressures of 20, 30, and 40 mTorr versus the rf power were plotted in Fig. 7. They increase with the rf power, suggesting larger ionization fractions at higher rf powers as expected due to an increase of plasma density. Figure 7 also shows the emission intensity ratios are larger at higher argon pressures under the same rf powers. At a rf power of 600 W, the ratio increases from less than 1 at 10 mTorr to about 1.8 at 40 mTorr.

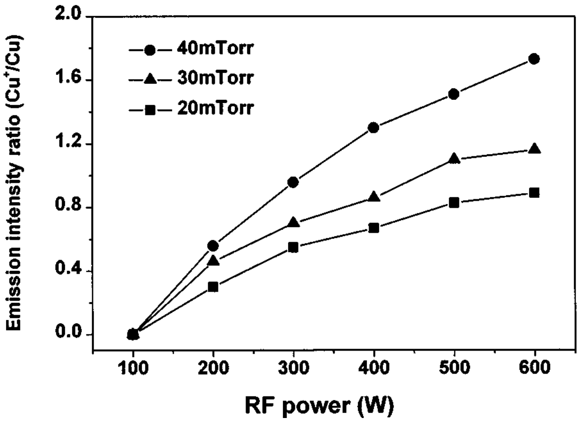

FIG. 7. The emission intensity ratio from Cu$^+$ ion to Cu neutral lines at 10, 20, and 40 mTorr as a function of rf power.

C. Biased quartz crystal microbalance measurements

To quantitatively determine the Cu neutral and Cu$^+$ ion fluxes in ionized sputtering of Cu, biased quartz crystal microbalance measurements were performed. Figures 8 and 9 show the Cu neutral and Cu$^+$ ion fluxes versus rf powers and argon pressures, respectively. The Cu neutral fluxes were measured by using the biased microbalance with a positive bias of 30 V. The Cu$^+$ ion fluxes were determined by subtracting the Cu neutral fluxes from the total Cu fluxes.[7] Figure 8 presents the Cu neutral fluxes at argon pressures of 20 and 40 mTorr as a function of rf discharge power. A sputtering power of 200 W was used. At 20 mTorr, the neutral deposition flux increases slightly with rf power from about 400 to 450 A/min at rf power of 600 W. A similar increase for the Cu neutral deposition flux also occurred at 40 mTorr. This increase is attributed to the antenna sputtering by the dc bias developed on the Cu antenna.[12] The bottom curve in

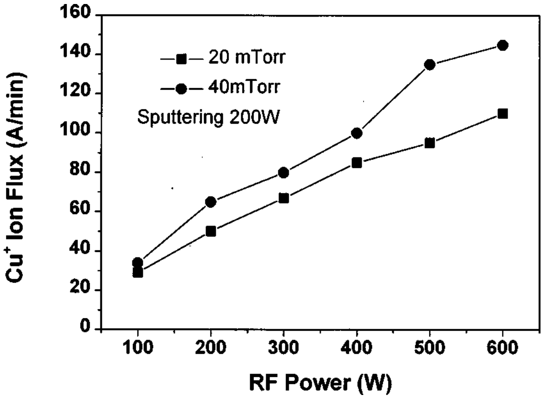

FIG. 9. Cu$^+$ ion fluxes at 20 and 40 mTorr vs rf power.

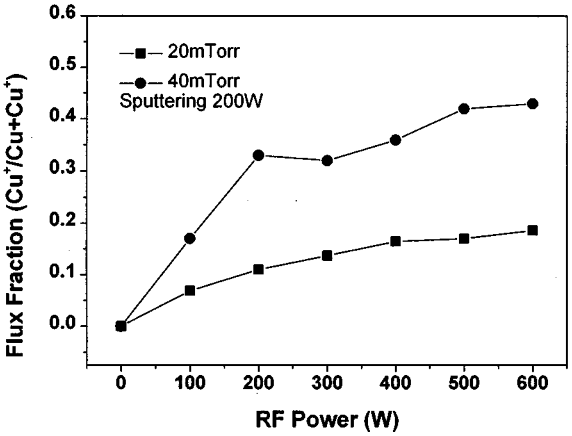

FIG. 10. Cu$^+$ ion flux ratio at 20 and 40 mTorr as a function of rf power.

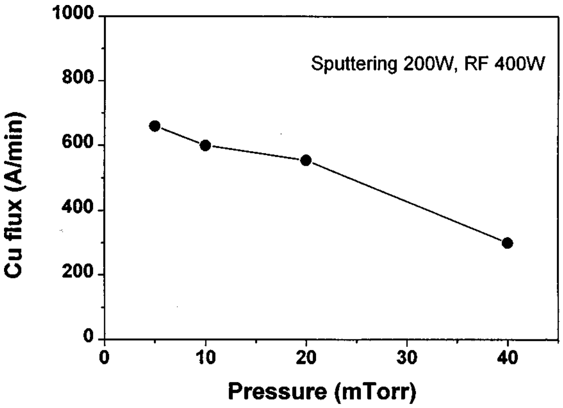

FIG. 11. The total deposition fluxes from Cu neutral and Cu$^+$ ions vs argon pressure at rf power of 400 W and sputtering power of 200 W.

Fig. 8 represents the Cu neutral fluxes from antenna sputtering versus rf power at an Ar pressure of 20 mTorr where no magnetron sputtering was used. As rf power increases, more Cu atoms are being sputtered out of the antenna.

The Cu$^+$ ion fluxes at 20 and 40 mTorr versus rf discharge power were plotted in Fig. 9. The Cu$^+$ ion fluxes monotonically increase with rf power, indicating that more Cu neutrals are ionized at high rf powers as the argon plasma density increases with rf power and more electrons become available to ionize the Cu atoms. Also shown in Fig. 9, the Cu$^+$ ion flux is larger at 40 mTorr than at 20 mTorr at the same rf power contrary to the Cu neutral fluxes as shown in Fig. 8, suggesting a higher degree of ionization of Cu at a larger argon pressure. Cu atoms are more likely to be ionized at higher argon pressures because of the increase in plasma density and residence time for Cu neutrals in the plasma region at higher argon pressures.[13]

A critical parameter in ionized sputtering is the ion flux fraction as defined in the introduction. The ion flux fraction is a key factor affecting conformal deposition and trench-filling capability and film properties during IPVD. It is desired to have as large an ion flux fraction as possile for better trench filling. From the measured Cu neutral and Cu$^+$ ion fluxes, the ion flux fractions can be determined at various rf powers and argon pressures. The Cu ion flux fractions at pressures of 20 and 40 mTorr versus rf power were shown in Fig. 10. It increases with rf power. At 20 mTorr, the ion flux fraction increases to about 20% at a rf power of 600 W. However, at 40 mTorr, the ion flux ratio increases to about 50% at the same rf power, suggesting a larger ion flux fraction at higher argon pressure. This is due to the increase of Cu$^+$ ion fluxes and a decrease for Cu neutral fluxes at higher pressures. At higher argon pressures, the plasma density and Cu residence time increases, thereby leading to higher ionization of Cu. This study suggests that a higher argon pressure than normal sputtering pressure (5 mTorr) is required to produce larger ion flux fraction in ionized sputtering.

The Cu$^+$ ion flux and Cu ion flux fractions increase with argon pressure as previously shown. However, the total fluxes of Cu neutrals and Cu$^+$ ions decrease with the argon pressure due to the significant reduction of Cu neutral fluxes at high pressure. Figure 11 presents the total fluxes of Cu at rf power of 400 W versus argon pressures. It shows a significant decrease of total Cu flux with argon pressure.

IV. SUMMARY AND CONCLUSION

Langmuir probe, optical emission spectroscopy, and biased quartz crystal microbalance measurements were used to study rf plasma plasma properties and Cu ionization in ionized sputtering. Magnetron-sputtered Cu vapor was injected into an Ar plasma excited by an internal rf antenna at a frequency of 13.5 MHz. An increase of plasma density with Ar pressure and rf discharge power was observed. The probe profiling shows a steeper ion density radial gradient at higher Ar pressures. The optical emission measurements indicate an increase of the intensity ratio from Cu$^+$ ion and Cu neutral lines with the rf power and argon pressure. The biased microbalance measurements confirms an increase of Cu$^+$ ion flux and the ion flux fraction with the rf power and argon pressure. However, the total Cu fluxes decrease significantly at a higher argon pressure.

This study indicates that a higher argon pressure than the normal sputtering pressure (1–5 mTorr) is necessary to achieve a larger Cu$^+$ ion flux fraction in ionized sputtering of Cu using an inductively coupled plasma. However, the total Cu flux decreases significantly at the high argon pressures. Also as the argon pressure increases, the argon plasma density becomes less uniform across the rf antenna. Such findings are important for choosing an operating pressure using ionized sputtering for semiconductor device manufacturing where higher ion flux fraction, larger deposition rate, and better uniformity across the wafer are required.

ACKNOWLEDGMENT

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